CHEMICAL ENGINEERING
ESSENTIALS FOR THE CPI PROFESSIONAL
www.chemengonline.com
July 2018

Maintaining Heat-Transfer Fluid Quality

page 32

Flow Measurement

Urban Mining

Predictive and Preventive Maintenance

Plant Expansions

Retrograde Condensation

Biosludge Drying

Facts at Your Fingertips: Packaging

Focus on Safety

Access Intelligence

CHEMICAL ENGINEERING HEAT-TRANSFER FLUIDS • FLOW MEASUREMENT VOL. 125 NO. 7 JULY 2018

CHEMICAL ENGINEERING
ESSENTIALS FOR THE CPI PROFESSIONAL

www.chemengonline.com

July 2018 Volume 125 | no. 7

Cover Story

In the News

Technical and Practical


32


14


18


38

52

24

27

Equipment and Services

Departments

Advertisers

Chemical Connections

Follow @ChemEngMag on Twitter

Join the *Chemical Engineering* Magazine LinkedIn Group

Visit us on www.chemengonline.com for more articles, Latest News, Webinars, Test your Knowledge Quizzes, Bookshelf and more

Coming in August

Look for: **Feature Reports** on Mixing; and Materials of Construction; A **Focus** on Software; A **Facts at your Fingertips** on Heat Transfer; **News Articles** on Achema 2018; and Pressure Relief; **New Products**; and much more

Cover design: Rob Hudgins

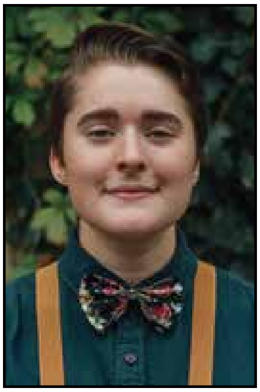
Editor's Page

The 2018 Chopey Scholarship

Since 1902, *Chemical Engineering* has been a leading source for practical technology information and news for the chemical process industries (CPI). Additionally, we strive to bring recognition to, and help advance the chemical engineering profession. With that in mind, in 2007, *Chemical Engineering* established the annual Chopey Scholarship for Chemical Engineering Excellence to offer assistance to a student who is working toward a degree in chemical engineering. The award is named after Nicholas P. Chopey, the magazine's former Editor-in-Chief, who devoted over 47 years of his professional career to this magazine.

The 2018 award winner

A.C. Carlton



Congratulations to this year's scholarship winner, A.C. Carlton. Carlton just completed an engineering co-op assignment at Kimberly-Clark in South Carolina where she was part of the Product and Process Improvement Team. This summer, she is working as an intern with Valero at its petroleum refinery in St. Charles, La., and is gaining experience in environmental engineering by working on projects designed to monitor emissions data for EPA compliance. She is enrolled as a junior for the fall semester at the University of Oklahoma and plans to graduate with a degree in chemical engineering in May 2020. Carlton is also a National Merit Scholar and she serves as the president of the oSTEM (www.ostem.org) chapter at the University of Oklahoma. We wish her all the best as she continues her education in chemical engineering.

About the scholarship

The scholarship is awarded to current third-year students who are enrolled in a fulltime undergraduate course of study in chemical engineering at one of the following four-year colleges or universities, which include Chopey's alma mater and those of our editorial staff: the University at Buffalo, University of Kansas, Columbia University, University of Virginia, Rutgers University and the University of Oklahoma.

The scholarship is a one-time award. The program utilizes standard Scholarship America recipient selection procedures, including the consideration of past academic performance and future potential, leadership and participation in school and community activities, work experience, and statement of career and educational goals.

More information about the award, including how to apply and how to contribute a donation, can be found at www.chemengonline.com/npcscholarship.

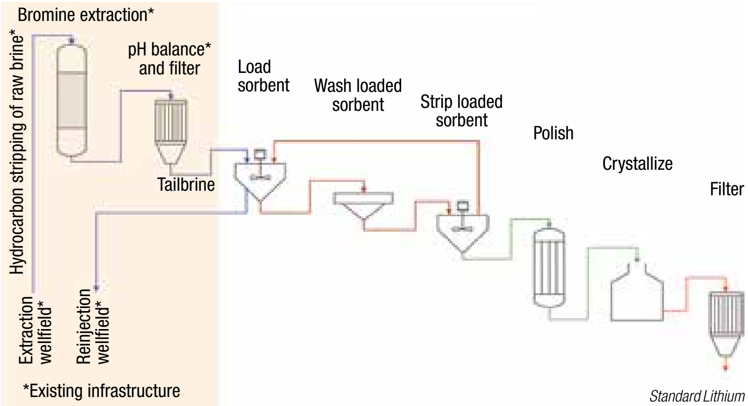
In this issue

Our July issue presents practical information on a wide variety of topics, including how to maintain the quality of heat transfer fluids in our Cover Story. Flow measurement and control is discussed in our two-part Feature Report, which includes an overview of selection criteria for measuring gas flow, and takes a deep dive into handling flow measurement of hazardous materials. Safety is also the focus of our Environmental Manager article on plant expansions and modifications. There is much more in our Newsfronts and in our regular departments. We hope you enjoy reading. ■

Dorothy Lozowski, Editorial Director

Extracting lithium from waste brine without ponds

The growing demand for lithium-ion batteries (LIBs) has created incentives to improve processes for extracting lithium from brine. Current Li-extraction processes involve pumping brine from underground into shallow pools for evaporation and concentration. But the traditional method has several significant limitations, including its requirement for large flat areas for ponds and a dry climate for evaporation. Further, the process takes over a year, only recovers 50% of available Li and comes with challenges in achieving high purities.



Standard Lithium

Standard Lithium Ltd. (Vancouver, B.C.; www.standardlithium.com) has developed a new process to overcome these limitations, while taking advantage of waste brine from another process as a resource. As a raw material, the process utilizes brine that has already been pumped from underground for the extraction of bromine. The company has partnered with Lanxess AG (Cologne, Germany; www.lanxess.com), which owns a bromine-extraction operation in southern Arkansas that is part of its flame-retardant chemicals business.

Standard Lithium developed a proprietary process that uses a solid ceramic adsorbent material with a crystal lattice that is capable of selectively pulling Li^+ ions from the waste brine after it has gone through the bromine-extraction step. The ceramic adsorbent materials are mounted in stirred-tank reactors that contain the brine. In the second step, the adsorbent releases the Li^+ ions for recovery.

"The technology piggybacks on all of the R&D work that has been conducted for LIBs," says Robert Mintak, CEO of Standard Lithium. "We are able to bolt this new adsorbent-based extraction process onto the back end of the bromine operation, where we have access to large volumes of brine that are already at the surface."

The Li-extraction process (diagram) takes advantage of the fact that the brine leaves the bromine process heated to approximately 70°C. This means that no additional energy is required, and the reaction kinetics for the adsorption are suitable, Mintak explains. The process is capable of reducing the time required for Li extraction from months (with the evaporation pools) to hours, and is capable of producing a high-purity LiCl solution for further processing in the battery industry.

The Li-extraction operation is currently operating as a "mini-pilot project," and a larger skid-mounted pilot plant will be deployed at one of Lanxess' Smackover Brine processing sites later this year.

Edited by:
Gerald Ondrey

FORWARD OSMOSIS

Last month, Bioindustrial Innovation Canada (BIC; Sarnia, Ont.; www.bincanada.ca), Forward Water Technologies (FWT; www.forward-water.com) and GreenCentre Canada (GCC; Kingston, Ont.; www.greencentrecanada.com) announced a private investment into FWT, supporting the commercial scaleup of a proprietary forward-osmosis technology. The initial technology behind FWT — a patented method using switchable solvents — originated from the laboratory of professor Philip Jessop at Queen's University (*Chem. Eng.*, November 2015, p. 13), and was further developed and launched as a spin-off company by GCC in 2012. This proprietary forward-osmosis system is said to be a highly energy-efficient process that has been successfully demonstrated for the treatment of many challenging high total-dissolved-solids wastewater streams that return fresh water for re-use in operations or discharge to either sewer or surface systems. FWT has been engaged in the commercial de-

(Continues on p. 8)

Making amine-based CO_2 absorbents more stable

Amine-containing solids have been investigated as promising adsorbents for CO_2 capture, but existing amine-containing adsorbents degrade by oxidation, making them unreliable for repeated CO_2 adsorption-desorption cycles over a long period. The low stability requires the continuous addition of fresh adsorbents, which significantly increases the cost of CO_2 capture.

Now researchers from the Korea Advanced Institute of Science and Technology (KAIST, Daejeon, South Korea; www.kaist.edu), led by professor Minkee Choi, have discovered that the very small amount of metal impurities — iron and copper — present in the amine accelerate the oxidative breakdown of the adsorbent. They propose the use of a chelator, which suppresses the activation of the impurities. Laboratory studies showed that the proposed method renders the adsorbent up to 50 times slower in its activation rate due to oxidation, compared with conventional polyethyleneimine (PEI)/silica adsorbents.

The researchers developed an extra-stable adsorbent by combining two strategies. First, PEI was functionalized with 1,2-epoxybutane, which generates tethered 2-hydroxylbutyl groups. Secondly, chelators were pre-supported onto a silica support to deactivate the metal impurities that catalyze amine oxidation. The resulting adsorbent exhibited a minor loss of CO_2 working capacity (8.5%), even after 30 days aging in O_2-containing fluegas at 110°C. This corresponds to a 50-times-slower deactivation rate than that of conventional PEI/silica, which exhibited a complete loss of CO_2 uptake capacity after the same treatment.

The researchers believe their work represents an important breakthrough for the commercial implementation of those adsorbents.

velopment of this technology and has brought it from the laboratory to almost commercial readiness.

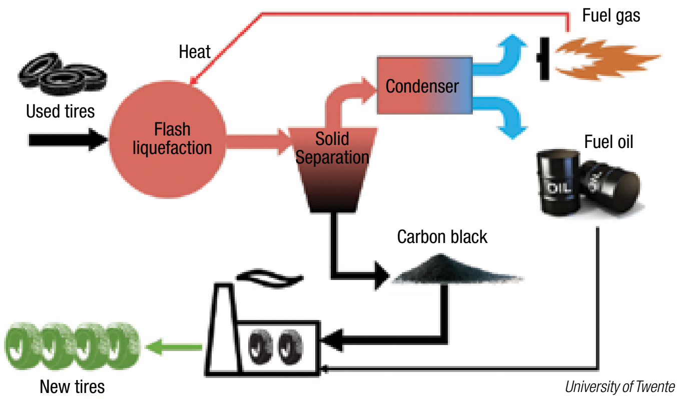
HYDROGEN STORAGE

Last month, the Catalysts business unit of Clariant (Munich, Germany; www.clariant.com) formed an alliance with Hydrogenious Technologies GmbH (HT; www.hydrogenious.net), to provide reliable, scalable and safe hydrogen supply solutions for a wide variety of applications. HT, a Friedrich-Alexander-University Erlangen-Nuremberg (FAU; Germany) spin-off, developed a means of transporting H_2 by chemically binding the molecules to liquid organic hydrogen carriers (LOHC). In the method, hydrogenation of dibenzyltoluene via Clariant's EleMax H catalyst allows hydrogen to be "stored," while its dehydrogenation with EleMax D "releases" H_2 on demand.

Non-explosive, non-toxic and of low flammability, the diesel-like hydrogen-bound compound is not classified a hazardous good, and remains in a useable and convenient liquid state through

(Continues on p. 9)

Fast-pyrolysis process recovers valuable materials from tires



University of Twente

Each year, more than 1 billion automobile tires are replaced worldwide. Together, the used tires contain 4.4 million metric tons of valuable products, such as carbon black, metals, fuels and chemicals. In an effort to recover these materials, the University of Twente (Enschede, the Netherlands; www.utwente.nl) and Continental Reifen Deutschland GmbH (Hannover, Germany; www.continental-reiden.de) are developing a new fast pyrolysis process that decomposes used tires at high temperatures in a matter of seconds.

In the process (diagram), tires are first pretreated to remove metal, fibers and textiles, and the rubber is milled to centimeter-sized granules. A screw feeder continuously feeds rubber granulate to a flash-liquification reactor, operating at a temperature of around 500°C. With residence times of a few seconds, the rubber is converted into pyrolysis gas and solids, explains project leader Gerrit Brem, professor of Thermal Engineering at Twente. The solids are recovered by a cyclone and the gas is condensed into fuel oil and non-condensable fuel gas.

Although the product yield depends on the operating conditions, Brem estimates that a given feed yields about 35 wt.% carbon black, 20 wt.% gas and 45 wt.% condensable oil.

"Unique is our fast heat transfer, resulting in a fast process of seconds, while conventional pyrolysis needs hours to complete the process," says Brem. "Because of the fast process, we expect — and have seen — a superior carbon black quality," he says.

The project, partially funded by the Materials Innovations Institute (M2i), aims for a design for a pilot plant and an optimum window for the process conditions. The plan is to scale up the process to demonstration scale in a follow-up project, says Brem.

The fast pyrolysis process is also capable of converting other waste streams into valuable materials. Examples are the recovery of fuels and minerals from paper sludge, carbon fibers from waste composites, or glass fibers from dismantled boats or wind turbines.

More scalable method for membrane separations

Membranes present a lower-energy alternative to distillation for the separation of hydrocarbons, but these processes are often limited by prohibitively expensive materials or intricate fabrication steps. Now, a team of researchers from Texas A&M University (TAMU; College Station, Tex.; www.tamu.edu) have developed a membrane-based separation technology for light olefins that is not only highly selective between similarly sized molecules, but also uses more scalable materials and methods.

The project centers around the separation of propylene and propane using a membrane composed of a zeolite-imidazolate framework (ZIF-8). ZIF-8 has been proven to effectively separate propylene and propane, due to its very small pore size. However, since polycrystalline ZIF-8 membranes are quite expensive, the team sought ways to decrease the cost of ZIF-8 membranes by drastically increasing propylene flux without adversely impacting its separation capabilities. The team integrated ZIF-90 into the membrane using a method called post-synthetic linker exchange (PSLE). Since ZIF-90's pore size is much larger than that of ZIF-8, strategic layering of ZIF-90 into ZIF-8 resulted in a membrane that reduced by four times the effective thickness of the ZIF-8 layer, thereby increasing propylene flux by four times, without compromising the selectivity of the membrane. This type of PSLE can be scaled up to commercial levels, says Hae-Kwon Jeong, associate professor of chemical engineering at TAMU.

Currently, the membranes have been demonstrated on 2.5-cm alumina discs, but the team is now working to prepare the membranes on pre-formed modules made of polymer hollow fibers, making the membrane fabrication step much more readily scalable, explains Jeong. The next steps of the research will focus on applying the membranes to the polymer modules and further reducing the thickness of ZIF-8 layers, while still maintaining optimal separation factors. According to Jeong, chemical companies in the U.S. and Japan have shown interest in further developing the technology.

Testing the limits of cryogenic distillation

CSIRO (Perth, Australia; www.csiro.au) and the University of Western Australia (www.uwa.edu.au) have developed a first-of-its-kind pilot plant for high-pressure cryogenic distillation processes. "Although cryogenic distillation is practiced in industry and there are some models that can be applied to cryogenic distillation, some gaps remain when it comes to high pressures. The facility will help to fill these gaps and better understand the fundamentals of distillation in temperature and pressure regions not previously explored," explains Nick Burke, CSIRO research scientist. The plant can operate at temperatures as low as –70°C and at pressures as high as 50 bars, with modifications enabling operation up to 100 bars. A reflux condenser fabricated by CSIRO will allow operation at even lower temperatures.

Currently, the pilot plant's work is aimed at processes for removing heavier hydrocarbons from natural gas prior to transformation to liquefied natural gas (LNG). "We can test different column internals and can operate under conditions that would not be considered in commercial facilities. In other words, we can push the limits of cryogenic distillation without the threat of deleterious process disturbances," adds Burke. "Understanding where these limits are will enable industry to better optimize their processes." He suggests that other applications that could benefit from high-pressure cryogenic pilot runs are air separation, CO_2 removal or noble-gas enrichment. The pilot column is 2 m high with a 50-mm diameter, and its maximum throughput is 20 L/min. The column can operate continuously and has autonomous process control.

Also, for floating LNG applications, the pilot plant's distillation column is designed to oscillate and tilt to mimic motion from wind and waves. "All of the methods for calculating distillation efficiency are based on the assumption of a vertical distillation column. The facility will allow the development of new, more accurate models and methods for tilted and moving columns," says Burke.

Antifouling membranes for single-step desalination

A filter for water purification that can replace, with a single step, the complex, time-consuming and multi-stage processes currently needed, has been developed by a team from CSIRO Manufacturing (Sydney, Australia; www.csiro.au), led by Dong Han Seo. The team has demonstrated water desalination via a membrane distillation (MD) process using a graphene membrane, called graphair. Water permeation is enabled by nanochannels of multilayer, mismatched, partially overlapping graphene grains.

As described in a recent issue of *Nature Communications*, Graphair is made by a chemical-vapor-deposition (CVD) process, and the film is then transferred onto a conventional MD membrane, such as polytetrafluoroethylene (PTFE). The CVD process takes place at ambient-air conditions using renewable soybean oil as the source for the graphene growth. This graphene film allows water to permeate through, but rejects salts and pollutants, such as surfactants and oils. Moreover, it prevents one of the great problems associated with desalination and filtration methods: fouling.

The team tested the pristine PTFE and permeable graphene-based membranes using saline solutions containing surfactants such as sodium dodecyl sulfate (SDS). As expected, significant fouling was evident for the pristine PTFE-based membrane. This membrane is able to process saline/SDS feed solution, but over 32 h there was a significant reduction in water flux from 40 $L/m^2/h$ to 14.2$L/m^2/h$. Salt rejection also decreased, from 100 to 97.1%.

In contrast, the permeable graphene-based membrane demonstrated stable and high water-vapor flux (50 $L/m^2/h$) and stable salt rejection (100%) over 72 h of operation under similar operating conditions. The superior antifouling performance of the graphair-coated PTFE was observed when a high concentration of oil compounds was included.

a broad temperature range of –39 to 390°C at ambient pressure. These factors allow considerably easier installation at industrial locations, as well as commercial and public fueling sites, even in close range of or within residential areas. It furthermore allows for the handling flexibility required to enable a widespread rollout of H_2 production from renewable power sources.

First commercial-scale units have been in operation since 2016.

IN SITU H_2 ANALYZER

Last month at Achema (June 11–15; Frankfurt am Main), Neo Monitors A/S (Skedsmokorset, Norway; www.neomonitors.com) launched what is claimed to be the world's first in situ optical analyzer for H_2. The LaserGas II SP H2 analyzer is based on traditional infrared (IR) tunable-diode-laser (TDL) absorption spectroscopy. "The H_2 molecule has for a long time been considered as non-absorbing in the IR region," says CEO Ketil Gorm Paulsen. "This is de facto incorrect, and by redesigning our analyzers we have achieved an unprecedented sensitivity down to the tiny absorption levels required to monitor H_2." With a response time of less than 2 s, the new device opens up new possibilities for process control in the oil-and-gas, petroleum-refining, steel and chemical industries, he says.

MONITORING MOLTEN STEEL

Steelworkers will be able to monitor in real time the temperature and chemical composition in molten metal furnaces, saving each steel plant up to £4.5 million/yr, thanks to a new laser-based technology developed by a Swansea University (www.swansee.ac.uk) spin-off company, Kubal-Wraith Ltd.

Currently in steelmaking, production is halted while disposable probes are immersed into the molten metal to measure temperature and take samples. This is inefficient as it takes up time, requires expensive probes and reduces productivity. In contrast, the new technology uses lasers, projected into the molten furnace, which monitor the contents continually. There is no need for disposable probes and production does not need to be stopped.

Szymon Kubal of Tata Steel and research fellow at Swansea University, says the new technology allows a laser beam to be projected into a

(Continues on p. 10)

molten furnace through a channel called a tuyère in the furnace wall. "We exploit the latest gas-injection techniques to protect the data channel." Full-scale trials have been carried out at Tata Steel UK.

The technology is also applicable to other metals, such as aluminium, copper and nickel. World Steel Association data indicate there are more than 1,000 molten-metal furnaces worldwide, which could see benefits in cost and productivity by using the new method of monitoring.

CROP PROTECTION

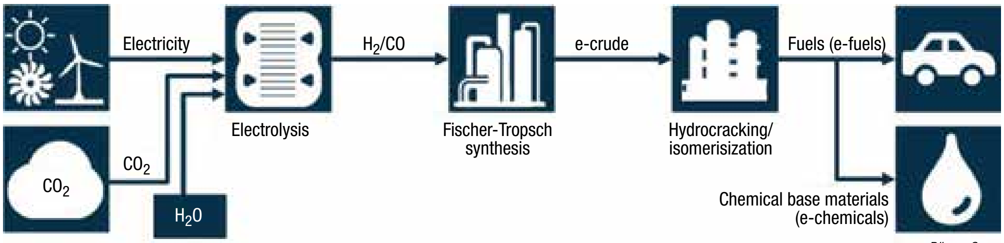
Traditional insecticides not only kill pests, they also endanger bees and other beneficial insects, as well as affect biodiversity in soils, lakes, rivers and seas. Now, a team from the Technical University of Munich (TUM; Germany; www.tum.de) has developed an alternative: A biodegradable agent that keeps pests away without poisoning them.

Professor Thomas Brück, head of the Werner Siemens Chair of Synthetic Biotechnology at TUM, and his team have found an insect repellent that is biodegradable and ecologically harmless. Sprayed onto plants, it works much like mosquito repellent, spreading a smell that keeps away unwanted insects. "With our approach, we are opening the door to a fundamental change in crop protection," says Brück. "Instead of spraying poison, which inevitably also endangers useful species, we deliberately merely aggravate the pests."

The TUM researchers were inspired by the tobacco plant, which produces cembratrienol (CBTol) in its leaves to protect itself from pests. Using synthetic biotechnology tools, Brück's team isolated the sections of the tobacco plant genome responsible for the formation of the CBTol molecules, then built these into the genome of *coli* bacteria. Fed with wheat bran, a byproduct from grain mills, the genetically modified bacteria now produce the desired active agent, which is separated from the fermentation broth by centrifugal separation chromatography.

Initial investigations indicate that the CBTol spray is non-toxic to insects, yet still protects wheat against aphids. Since it is biodegradable, it does not accumulate. In addition, the bioactivity tests showed that CBTol also has an antibacterial effect on gram-positive bacteria, so it can also be used as a disinfectant spray against pathogens. ❑

The first commercial power-to-liquids plant planned



Pörner Group

Nordic Blue Crude A/S (NBC; Porsgrunn, Norway; www.nordicbluecrude.no) intends to build the first power-to-liquids (PtL) plant in the Norwegian industrial park of Heroya, located 140 km southeast of Oslo. Although the date for the startup has not yet been released, the plant will produce 8,000 ton/yr of "e-crude" from CO_2, water, and 20 MW of hydroelectric power. The crude oil will then be refined into "e-waxes and e-fuels." Engineering services for the project are being performed by EDL Anlagenbau GmbH (EDL; Leipzig, Germany; www.edl.poerner.de), a 100% subsidiary of the Pörner Group (Vienna, Austria; www.poerner.at), and Sunfire GmbH (Dresden, Germany; www.sunfire.de).

The first step of the PtL process (diagram) is the renewable generation of synthesis gas (syngas), either by a steam electrolysis and CO_2 conversion by the reverse water gas shift (RWGS) reaction, or from a direct electrochemical transformation by co-electrolysis. The syngas is then converted to liquid hydrocarbons (e-crude) via Fischer-Tropsch (F-T) synthesis. The e-crude can then be further processed into high-value fuel components (e-fuels) and chemical base materials (e-chemicals).

The plant will use Sunfire's solid oxide electrolysis cell (SOEC) technology for steam electrolysis. Most of the CO_2 will be supplied from the industrial byproduct of a neighboring site. The carbon source will be supplemented with CO_2 captured from air using the DAC (Direct Air Capture) technology of Climeworks (Hinwill/Zurich, Switzerland; www.climeworks.com; for more details about this technology, see *Chem. Eng.*, July 2017, p. 7). For the RWGS stage the F-T synthesis, the use of commercially available catalysts is envisaged. A downstream fractionation unit will separate the e-waxes and e-fuels.

An inexpensive, renewable aerogel shows promise for handling oil spills

Many methods have been developed to remove spilled oil and organic pollutants from water, but they all entail shortcomings, such as low absorption ability and poor buoyancy. Now, a team from the School of Light Industry and Chemical Engineering, Dalian Polytechnic University (Dalian, China; www.dipu.edu.cn), and Zhejiang Ji-Hua Group Co. (Hangzhou, China; www.jihuadyes.com) has prepared a low-cost, ultralight absorbent aerogel from renewable corn straw and filter paper with good performance and high absorption capacity. According to the team, the corn-straw-based spongy aerogel could serve as a good oil-absorbing material.

To make the aerogel, raw corn straw is first added to an aqueous sodium hydroxide solution, and stirred for 4 hours at 30°C. Next, the pH is adjusted to 7 by the addition of HCl. After thorough filtration and washing with deionized water, the filtered corn straw particles are dried for 12 hours at 60°C. Small filter paper fragments are then mixed with the treated corn straw, and dispersed with a high-shear emulsifer. The suspension was then frozen at –25°C for 12 hours, followed by freeze drying at –55°C for 36 hours, resulting in a spongy aerogel. The hydrophobicity of the aerogel was improved by a silanation reaction, using chemical-vapor deposition.

Because of the aerogel's ultralow density, high porosity and hydophobicity, the aerogel is said to exhibit a "remarkable" absorption capacity for both crude oil (36 g/g) and common organic solvents, including carbon tetrachloride (45 g/g), dimethyl sulfoxide (24 g/g) and N,N-dimethylformamide (45 g/g). The team says this could point the way for the design of efficient absorbents for oil spills and organic pollutants. ■

LINEUP

AKZONOBEL
ARKEMA
ASCEND PERFORMANCE MATERIALS
BASF
CHANDRA ASRI
DUPONT
HEXCEL
HITACHI CHEMICAL
INEOS
MILLIKEN
OXEA
OXITENO
POLYONE
SADARA
TOYO
UMICORE
VEOLIA

Plant Watch

Arkema to expand polyamide powder production capacity in France

June 6, 2018 — Arkema (Colombes, France; www.arkema.com) plans to expand its global production capacity for specialty polyamide powders marketed under the brand name Orgasol by more than 50% at its Mont, France site. The investment for this project is around €20 million. The new capacity is expected to come onstream in the second half of 2019.

Ineos moves forward with Gulf Coast ethylene-oxide project

June 6, 2018 — Ineos (Rolle Switzerland; www.ineos.com) is moving forward with the next stage of its project to construct an ethylene-oxide (EO) and ethylene-oxide-derivatives (EOD) facility on the U.S. Gulf Coast. It is planned that the plant will be operational in 2022. The plant is expected to produce around 270,000 metric tons per year (m.t./yr) of EO, with ethoxylate derivative capacity onsite.

DuPont invests $400 million in Tyvek expansion in Luxembourg

June 6, 2018 — DuPont Safety & Construction, a business unit of DuPont (Wilmington, Del.; www.dupont.com), announced plans to invest more than $400 million to expand capacity for the manufacture of Tyvek nonwoven materials at its facility in Luxembourg. The production expansion, which will add a new building and third operating line at the site, is scheduled to start up in 2021.

Toyo wins engineering contract from Chandra Asri

June 1, 2018 — Toyo Engineering Group (Chiba, Japan; www.toyo-eng.co.jp) was awarded a contract by PT Chandra Asri Petrochemical Tbk (CAP; Jakarta, Indonesia; www.chandra-asri.com) that involves the construction of a 1-butene production unit with capacity of 43,000 m.t./yr and a methyl *tert*-butyl ether unit with capacity of 127,000 m.t./yr, as well as an enclosed ground flare with capacity of 220 m.t./h. The units will be constructed inside CAP's existing petrochemical complex in Cilegon, Java, Indonesia. The plant is scheduled for completion in 2020.

Umicore selects Poland to build new cathode-materials plant

June 1, 2018 — Umicore N.V. (Brussels, Belgium; www.umicore.com) has selected Nysa, Poland as the location for a production plant to manufacture battery cathode materials for the European automotive market, part of a €660-million investment program announced earlier this year. The plant is expected to start commercial production in late 2020.

BASF opens electronic-grade sulfuric-acid plant in China

June 1, 2018 — BASF SE (Ludwigshafen, Germany; www.basf.com) started operations at a new electronic-grade sulfuric-acid plant in Jiaxing, China, to serve the country's growing semiconductor industry. Furthermore, the plant has simultaneously started an expansion phase to double the production capacity before the completion of the facility. The expansion phase is expected to be operational by the end of the year.

Arkema plans to double thiochemicals production at Beaumont site

June 1, 2018 — Arkema has confirmed plans to double production capacities at its Beaumont Thiochemicals site in Texas. Subject to a final investment decision, expected to be made at the end of 2018, the new units should come onstream by the summer of 2021. The expanded capacity would supply sulfur derivatives for the new methionine-hydroxy-analogue production unit to be built by Novus International, Inc.

Ascend Performance Materials to expand adiponitrile production capacity

June 1, 2018 — Ascend Performance Materials, LLC (Houston; www.ascendmaterials.com) announced plans to expand its adiponitrile (ADN) capacity through 2022. In total, Ascend will grow its ADN capacity by 220,000 m.t. by 2022 to meet increasing demand. Ascend completed its first expansion of 50,000 m.t. at the end of 2017. An additional 40,000-m.t. expansion will be completed by the end of 2018, with plans for an additional 180,000 m.t. of capacity to be realized by 2022.

Oxea achieves mechanical completion of Bay City propanol plant

May 29, 2018 —Oxea GmbH (Monheim am Rhein, Germany; www.oxea-chemicals.com) reached mechanical completion of a new propanol production unit at its Bay City site in Texas. With a nameplate capacity of 100,000 m.t./yr of propanol, the new facility is now being commissioned and is scheduled for commercial production in the third quarter of 2018.

AkzoNobel to expand production of organic peroxides in India

May 24, 2018 —AkzoNobel Specialty Chemicals (Amsterdam, the Netherlands; www.akzonobel.com) broke ground on a project to upgrade its organic peroxides facility in Mahad, India. The €4-million investment will increase capacity by 80% and is expected to be completed by the end of 2018. The company is also investing in a monochloroacetic acid project in Gujarat, due to start production in 2019.

Look for more latest news on chemengonline.com

Mergers & Acquisitions

Hitachi Chemical signs licensing agreement with Silatronix

June 5, 2018 — Hitachi Chemical Co. (Tokyo, Japan; www.hitachi-chem.co.jp) and Silatronix, Inc. (Madison, Wisc.; www.silatronix.com) have entered into a licensing agreement concerning the manufacturing and use of Silatronix's patented organosilicon compounds as an electrolyte material in additives for Hitachi Chemical's anode materials for lithium-ion batteries. Hitachi Chemical will continue to evaluate battery performance to consider the commercialization of the additive.

Hexcel forms aerospace composites joint venture in China

June 1, 2018 — Hexcel Corp. (Stamford, Conn.; www.hexcel.com) has formed a joint venture (JV) with aerospace parts manufacturer Future Aerospace to establish a materials-testing laboratory in China. The new JV, Shanghai Future Aerospace Hexcel Commercial Composite Testing Ltd., will be located in Lingang, China. Operations at the new site are expected to begin in September 2018.

PolyOne acquires composite specialist PlastiComp

June 1, 2018 — PolyOne Corporation (Cleveland, Ohio; www.polyone.com) has acquired PlastiComp, a producer of specialty composites and advanced engineered materials based in Minnesota. Founded in 2003, PlastiComp has steadily grown through its ability to replace metal and lightweight products with complex long-fiber-technology (LFT) composite formulations.

Sadara and Veolia sign MOU for sustainable utilities

May 30, 2018 — Sadara Chemical Co. (Jubail, Saudi Arabia; www.sadara.com) has signed a memorandum of understanding (MOU) with Veolia (Paris, France; www.veolia.com) to construct a sustainable central utilities facility in PlasChem Park adjacent to the Sadara Chemical Complex. The utilities facility will incinerate industrial wastes and recover the heat to produce a usable steam byproduct.

DuPont divests Alginates business to JRS Group

May 29, 2018 — DuPont Nutrition & Health has received approval from the European Commission to divest its Alginates business unit to JRS Group, a manufacturer of functional additives from plant-based raw materials. The divestiture is expected to close in the third quarter of 2018. The acquired business comprises pure and buffered alginates, a specific portfolio of pectin-alginate blends and an associated production site in Landerneau, France.

Milliken and Oxiteno enter co-manufacturing agreement

May 25, 2018 — Milliken & Co. (Spartanburg, S.C.; www.milliken.com) and Oxiteno (São Paulo, Brazil; www.oxiteno.com) have entered into a multi-year co-manufacturing agreement, which will utilize Oxiteno's new alkoxylation unit in Pasadena, Tex. The facility, expected to open in the second quarter of 2018, incorporates alkoxylation assets and technologies, and has a nameplate capacity of 170,000 m.t./yr. ■

Mary Page Bailey

Urban Mining Offers Good Prospecting

As the volume of discarded electronic gadgets grows, efforts are underway to recover the precious resources trapped inside

IN BRIEF

PLANT EXPANSIONS

EMERGING PROCESS TECHNOLOGY

THE GROWING NEED FOR RECYCLING

It should come as no surprise that the fastest growing part of the world's domestic waste stream is e-waste — discarded products with a battery or plug (see box on p. 16). Because e-waste contains valuable resources, efforts around the word to collect and recycle such waste — so-called urban mining — are growing. But it is more than simply mining for profit. "Waste electrical and electronic equipment (WEEE, or e-waste) is a complex mixture of materials; it is a source of secondary raw materials, including precious metals, but, at the same time, it also contains hazardous substances that give rise to environmental and health problems if not adequately managed," according to WEEE Forum (Brussels, Belgium; www.weee-forum.org).

Hereaus Precious Metals

FIGURE 1. Hereaus employee working on the new furnace at the Wartburg, Tenn. facility

It was with this in mind — that e-waste is hazardous waste — that E.U. legislators adopted directive 2002/7967/EC on waste electrical and electronic equipment (WEEE) back in 2002. At that time, there was no specialist WEEE de-polluting and recycling industry to speak of. There were few WEEE designated collection facilities. Citizens were not aware of the hazards associated with discarding WEEE, and no targets or responsibilities were in place, according to the WEEE Forum. Since then, progress has been made, but there is still a lot of work to be done.

Plant expansions

Umicore N.V. (Brussels, Belgium; www.umicore.com) operates one of the world's largest metals-recycling plants in Hoboken, Belgium. This plant recovers precious (Ag, Au, Pt, Pa, Pd, Rh, Ir, Ru) and specialty metals (In, Se, Te) from a wide variety of materials from over 200 complex input streams from around the world (*Chem. Eng.*, April 2015, pp.18–23). The company recently invested €100 million to expand its capacity by 40%, and is now ramping up from 350,000 metric tons per year (m.t./yr) to 500,000 m.t./yr.

Meanwhile, last May, Hereaus Precious Metals, a global business unit of the Hereaus Group (Hanau, Germany; www.hereaus.com) inaugurated a 30,000-ft^2 expansion of its precious-metals recycling plant in Wartburg, Tennessee. The multi-million dollar expansion project, which began in the spring of 2016, increases the plant's pre-processing capacity to meet growing demand from the chemical, electronics, automotive and jewelry industries. Established in 2004, the Hereaus Wartburg plant is a treatment and storage facility for both hazardous and non-hazardous materials containing precious metals. The company says it has made significant investments in new recycling technologies and capabilities, including specialized converters, furnaces (Figure 1) and processing equipment (Figure 2). One of the newest innovations at Wartburg is its pyrometalurgical recycling converter, which uses a highly specialized process to recover precious metals faster and more economically, says the company. The converter will primarily focus on the recovery of platinum-group metals. "Processing and recovering precious

metals has become extremely complex. But today we have a state-of-the-art facility and a world-class team of talented employees at Wartburg to meet our customers' recycling challenges," said Andre Christl, president of Heraeus Precious Metals, during the ribbon-cutting ceremony.

The 358-year-old company operates a global network of trading offices, processing facilities and recycling sites in Germany, Switzerland, China, South Africa, India, Hong Kong and the U.S.

In March, the Alba Group (Berlin, Germany; www.alba.info) opened its Hong Kong WEEE-Park, a new facility for processing and recycling WEEE. Located in the city's EcoPark north of the airport, the new processing plant is said to be the most modern in all of Southeast Asia, and the biggest combined facility for processing such a wide range of WEEE in the world. Initially, the plant will have a capacity of 30,000 m.t./yr, with the option to expand to 56,000 m.t./yr.

In addition to the construction and operation of the plant, Alba Group's contract — the largest in its history — includes the development and operation of a city-wide collection system with five satellite collection centers and a fleet of heavy goods vehicles (HVGs) over 10 years. The facility will handle "legally regulated" electronic waste, comprising large household devices, including air conditioners, refrigerators, television sets, washing machines and computers.

Hereaus Precious Metals

FIGURE 2. Hereaus workers discussing the production process at the Wartburg plant

Emerging process technology

While traditional processing plants continue to expand, efforts are also underway to develop new processes for recovering valuable resources from e-waste. For example, Mint Innovation (Auckland, New Zealand; www.mintinnovation.co) is developing a hybrid approach that combins hydrometallurgy and biotechnology to recover gold from printed circuit boards (PCBs). "We take comminuted PCBs, put them through a leach process, and contact the resulting pregnant lixiviant with select microorganisms," explains chief scientific officer Ollie Crush. "These microbes are able to specifically biosorb precious metals, like gold, and enable both the purification and concentration of metal in one step. The process runs at ambient pressure and temperature and doesn't utilize cyanide."

THE GROWING NEED FOR RECYCLING

Umicore

In 2016, 44.7 million metric tons (m.t.) of e-waste — everything from end-of-life refrigerators and television sets, to solar panels, mobile phones and computers — was generated globally, according to The Global E-waste Monitor 2017, which was published last December. The report, compiled by the United Nations University (UNU; Bonn, Germany; www.unu.edu), the International Telecommunications Union (ITU; Geneva, Switzerland; www.itu.int) and the International Solid Waste Association (ISWA; Vienna, Austria; www.iswa.org), shows an 8% increase in e-waste generation compared to 2014, and experts foresee a further 17% increase to 52.2 million m.t. by 2021 — the fastest growing part of the world's domestic waste stream, says UNU.

The report also says that only 20% of 2016's e-waste is documented to have been collected and recycled, even though the waste contains rich deposits of gold, silver, copper, platinum, palladium and other materials — with a conservative estimated value $55 billion, says UNU. Most of the 2016 waste (76%, or 34.1 million m.t.) actually ended up being incinerated, thrown into landfills, or remains stored in households.

Europe (including Russia) is the second-largest generator of e-waste per inhabitant, with an average of 16.6 kg per inhabitant, but Europe also has the highest collection rate (35%), according to UNU. This could be due to the progress made since 2002, when the E.U. legislators adopted directive 2002/7967/EC on waste electrical and electronic equipment (WEEE).

According to the WEEE Forum (Brussels, Belgium; www.weee-forum.org), quantities of WEEE collected by the producer responsibility organizations (PROs) rose from 292,550 m.t. in 2002 to 2.1 million m.t. in 2016. However, recycling rates for most critical raw materials (CRMs) have remained low over the last 15 years, says WEEE Forum. "The recycling rate for CRMs, such as rare earth elements, is currently estimated at less than 1%," WEEE Forum reported in its 15-year anniversary brochure, published in 2017.

One initiative to capitalize on this lost resource is the development of a centralized database, with easy access to primary and secondary raw materials data on a single platform. Launched in January 2018, The Urban Mine Platform (www.urbanmineplatform.eu) was created by 17 partners in the ProSUM project (Prospecting Secondary Raw Materials in the Urban Mine and Mining Wastes). The database reveals the amount of valuable materials recovered or lost in the E.U.'s scrap vehicles, batteries, computers, phones, gadgets, appliances and other high-tech products discarded every year. "Three years in the making, this consolidated database is the world's first 'one stop shop' knowledge data platform on CRMs in waste products — easy to access, structured, comprehensive, peer-reviewed, up-to-date, impartial, broad in scope, standardized and harmonized, and verifiable, says," Pascal Leroy, secretary general of the WEEE Forum and ProSUM project coordinator.

According to the ProSUM consortium, a smartphone contains around 40 different CRMs, with a concentration of gold 25 to 30 times that of the richest primary gold ores. Furthermore, mining discarded high-tech products produces 80% less CO_2 emissions per unit of gold, compared with primary mining operations. ❑

"Because gold accounts for approximately half of the metallic value of PCBs, this is our process' primary target, followed by palladium and copper," says Crush. The aim for Mint is to commercialize a low-CAPEX, low-OPEX process that can be deployed economically at a range of scales, explains Crush. "This will enable individual cities or regions to be able to capture value from locally-generated WEEE, as opposed to landfilling or exporting it. In comparison to large-scale specialized smelters, this distributed approach will allow aggregators of WEEE to achieve higher returns, as well as shorten payment timeframes and reduce uncertainty. It is also hoped that our process can be deployed in developing nations in order to displace rudimentary (and hazardous) small-scale processing," he says.

Since December 2017, the company has been operating a pilot plant, located at Level Two (www.leveltwo.tech) in Auckland. The pilot plant can process tens of kilograms of ground PCBs at a time in 100-L batches, and Crush envisages a semi-continuous process at commercial scale. Mint has partnered with Remarkit Solutions Ltd. (Auckland, New Zealand; www.remarkit.co.nz) to build a demonstration plant that will recover precious metals from up to 200 m.t./yr of scrap PCBs. Construction is slated for early 2019, with commissioning taking place in stages over that year, says Crush.

Last April, the world's first e-waste microfactory started up at the University of New South Wales (UNSW; Sydney, Australia; www.unsw.edu.au). Using technology developed at UNSW's Center for Sustainable Materials Research and Technology, the microfactory transforms components from e-waste, such as discarded smartphones, laptops and printers, into valuable materials for re-use.

The discarded devices are first placed into a module to break them down. The next module may involve a special robot for the identification of useful parts. Another module then involves using a small furnace that transforms these parts into valuable materials by using a precisely controlled temperature process.

These transformed materials include metal alloys (copper-tin, for example) and a range of micromaterials. The micromaterials can be used in industrial-grade ceramics, while the specific quality plastics from computers, printers and other discarded sources can be put through another module that produces filaments suitable for 3-D-printing applications. The metal alloys can be used as components for new or existing manufacturing processes, says UNSW.

"These microfactories can transform the manufacturing landscape, especially in remote locations where typically the logistics of having waste transported or processed are prohibitively expensive," says professor Veena Sahajwalla. "This is especially beneficial for the island markets and the remote regions of the country."

The technology was developed with support of the Australian Research Council and is now in partnership with a number of companies, including e-waste recycler TES (Singapore) Pte Ltd. (www.tes-amm.com) and mining manufacturer Moly-Cop (Waratah, Australia; www.molycop.com).

Alba Group

FIGURE 3. The new recycling facility in Hong Kong is said to be the largest of its kind in the world

In another effort towards zero-waste solutions for e-waste, Ronin8 Technologies Ltd. (Richmond, B.C., Canada; www.ronin8.com) has developed a process to separate metal from the nonmetal matrix of PCBs using physical processes, as an alternative to chemical or thermal treatments that destroy the nonmetallic resources. The remaining nonmetal fraction, which usually end up in the landfills, can also be recovered in the process for secondary usage, and thus provides a closed-cycle solution for e-waste recycling, says the company.

Ronin8 has been working with the Ph.D. candidate Amit Kumar and professor Maria Holuszko at the Urban Mining Innovation Center (UMIC) at the University of British Columbia (UBC; Vancouver; www.ubc.ca) for one year to characterize and process the obtained nonmetal fraction for the suitability for secondary use. Most e-waste recycling firms focus on recovering useful metals like gold, silver, copper and palladium, which can be used to manufacture other products. But nonmetal parts like fiberglass and resins, which make up the bulk of cellphones' PCBs, are generally discarded because they're less valuable and more difficult to process. They are either fed to incinerators or landfilled, where they can leach hazardous chemicals into groundwater, soil and air.

Holuszko and Kumar developed a process that uses gravity separation and other simple physical techniques to process cellphone fiberglass and resins in an environmentally neutral fashion. "The separated fiberglass can then be used as a raw material for construction and insulation. In the future, if we can find a way to improve the quality of the recycled fiberglass, it may even be suitable for manufacturing new circuit boards," says Kumar.

Meanwhile, researchers from Rice University (Houston; www.rice.edu) and the Indian Institute of Science (Bangalore; www.iisc.ac.in) have shown that another possible way to separate the materials in PCBs is by cryogenic milling into nanopowders. Described in a March 2017 issue of *Materials Today*, the process uses a ball mill, with argon atmosphere operated at 154K, to break down the waste into nano-sized particles of metals, oxides and polymers. Because of the small size of the resulting dust (20–100 nm), the different powders behave as separate phases, which can then be easily separated. ■

Gerald Ondrey

The Changing Face of Maintenance

Data analytics, connectivity and machine learning are helping processors maintain facilities in a more cost-efficient way

IN BRIEF

Modern technologies, such as smart sensors, data analytics, the industrial internet of things (IIoT) and machine learning, are changing the face of maintenance to help processors ensure they are spending their maintenance dollars in the wisest way possible. While it may seem like a data-centric maintenance practice will require a lot of high-tech equipment, experts say chemical processing facilities likely have some of the necessary pieces already in place, and a variety of approaches allows users to go as light or as heavy as is desirable (Figure 1).

AspenTech

FIGURE 1. "The idea of using connectivity and analytics for predictive maintenance allows users to go all in and look at reliability of the asset and the business as a system," says Robert Golightly of AspenTech. "If you have a system view of what's going on, you can address the little vulnerabilities, as well as the big upsets, before they negatively impact the process or the business."

The evolution of maintenance

"For a long time there was only reactive maintenance," says Jerry Browning, senior advisor for enterprise maintenance, North America, with IFS (Itasca, Ill.; www.ifsworld.com). "Something was only fixed when it broke and maintenance techs ran from fire to fire. Then came preventive maintenance, which used available information on life expectancy of a class of assets to get ahead of the curve and provide maintenance on a routine schedule."

There are pros and cons associated with preventive maintenance. "The good part is preventive maintenance can save 10 to 15% in downtime and repair costs compared to reactive maintenance. The downside is preventive maintenance isn't always cost effective because it is hours- or cycle-driven and maintenance is performed based upon tribal knowledge or OEM [original equipment manufacturer] recommendations on equipment that may not be in danger of failure."

Nathan Hedrick, flow product marketing manager with Endress+Hauser (Greenwood, Ill.; www.us.endress.com) explains the preventive maintenance philosophy with the following example: "A flowmeter may have had a problem with buildup that shut down the operation. As a result, the maintenance team creates a preventive maintenance schedule that cleans every flowmeter in that unit semi-annually to avoid future shutdowns. While it may prevent future failure, it could also be that the shutdown was an anomaly. If that's the case, maintenance is spending a lot of time and resources to inspect and clean meters that are fully functional."

Not only does this squander time and money, but it "treats the symptom instead of the disease," says Hedrick. "The symptom would be that the meter caused the process to shutdown, but the disease could be an excursion in temperature that caused the media to solidify and trip up the meter."

However, had the maintenance team had access to diagnostics, they may have been able to address the problem before failure. Further, if they had access to process data, they may have been able to see the trend and identify the root cause of the problem instead of simply addressing the symptom at the back end, says Hedrick.

Enter the next evolution of maintenance: predictive maintenance, which was initially based upon the actual condition of the equipment it-

Yokogawa

Emerson

FIGURE 2. Yokogawa's SensPlus Buddy provides visual transmission video calls and augmented reality, as well as information sharing by sending images and texts. This improves the efficiency of maintenance work, reduces losses due to mistakes and facilates safe and worry-free plant maintenance

FIGURE 3. Emerson's Plantweb Insight software apps analyze the data from specific assets, such as pumps, and provide predictive maintenance information, such as imminent failures

self as determined by diagnostic data collected from hand-held devices or automated instruments. "Oil analysis, vibration analysis, acoustics, thermography and the like provide a very good indication of how well a piece of equipment is performing or where certain parts of the process are degrading so problems can be addressed before failure," notes IFS's Browning.

The most current iteration of predictive maintenance incorporates equipment health status, process data and analytics to identify abnormal situations and then calls for the necessary maintenance actions to avoid failures and downtime, says Brian Joe, wireless product manager for Emerson Automation Solutions (Shakopee, Minn.; www.emerson.com). "Predictive maintenance ensures companies spend resources more effectively on issues as they begin to happen," says Joe. "Predictive maintenance doesn't waste resources replacing things before it is necessary. This allows some asset-maintenance schedules to be extended. It also eliminates some failures that preventive maintenance would not resolve."

While it is not likely that predictive maintenance will wholly replace preventive techniques, there is a move-

Emerson

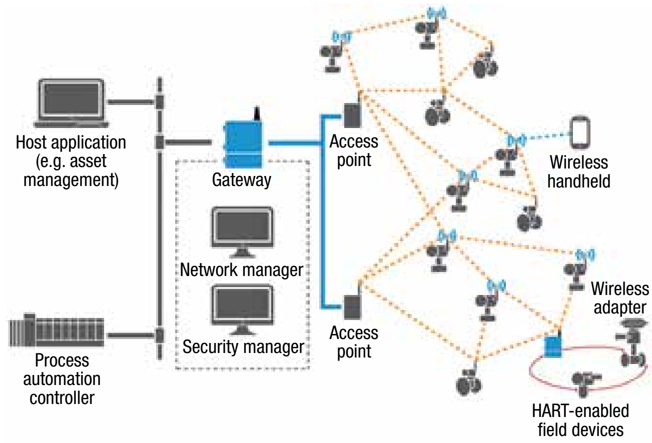


FIGURE 4. WirelessHART sensors and networks provide a low cost way to gather the data required for effective predictive maintenance

SAP

FIGURE 5. Building an intelligent enterprise includes connectivity and integration between all the components, processes and organizations so that processors may leverage the information in multiple ways allowing actions to be performed as part of the overall business process

ment toward blending the two methodologies. "We probably won't see any situations where preventive practices are eliminated entirely," explains Joe. "For some asset classes, doing things on a schedule makes sense and some low-criticality assets will be allowed to run to failure because the potential impact is so low."

He continues: "The extent to which predictive practices are deployed will increase as more diagnostic instruments become available at lower costs combined with simpler and cheaper data collection and analytics platforms. We're seeing that happen right now."

The variety and number of asset classes covered by predictive practices is increasing, but many actions will still be driven by a schedule, he explains. "Even with predictive maintenance practices in place, plants will likely still perform periodic maintenance on pumps, heat exchangers and similar equipment, even when predictive intelligence does not indicate an issue."

Masahara Aonuma, manager, lifecycle service business division, IA Systems and Service Business with Yokogawa (Sugar Land, Texas; www.yokogawa.com), agrees that a combined approach is a good one. "The benefits of preventive maintenance are finding faults and improving asset condition. The benefits of predictive maintenance are reducing system downtime, optimizing part lifecycle and improving productivity, which is possible because prediction is guided by measured data and enabled by technological innovation [Figure 2], such as IIoT and artificial intelligence," says Aonuma. "Both services are important elements and the two maintenance techniques are being incorporated into one maintenance practice to offer the best lifecycle execution plan. In order to maintain stable and continuous operation, effective lifecycle planning must be implemented to maximize system performance."

Today's maintenance toolbox

According to Endress+Hauser's Hedrick, doing this type of predictive maintenance requires instrument and system monitoring, as well as analysis of data. "To simplify, we say there are Three A's to the process: Acquire, Accumulate and Assess." To acquire the necessary data, there must be a way to get the data out of the process, which relies upon smart instrumentation and some sort of digital communication system or multiple analog signals to retrieve and move the data from the instruments. There must also be a place to accumulate that data, which may fall to the historian. "Many processors already have a historian on their control system, which means they may not need to add infrastructure, but rather allocate more memory to accumulate the process data," he says.

Another option might be sending it to the cloud via an edge gateway or device (which sits on the "edge" of the network and serves as a seamless integration point between IIoT and the cloud). From there, the data must be assessed, turned into useful information and delivered to the appropriate person. This is the part of the equation that varies the most. Some processors may choose to use software designed for this purpose. Others may choose to partner with an instrumentation, software or automation supplier offering this service.

For today's predictive maintenance analytics, modern technologies often come into play, says Yokogawa's Aonuma. "The latest technologies, such as IIoT, artificial intelligence, machine learning, environmental diagnosis and compact wireless sensors, increase prediction accuracy," he says.

Here's how they all work together:

Data collection. The first step to an effective predictive maintenance plan is gathering the data necessary to make intelligent decisions, says Emerson's Joe. By obtaining more relevant realtime data, such as pressure, temperature and vibration, on important assets, reliability and maintenance personnel can take more informed actions (Figures 3 and 4).

If this is all new, says Hedrick, the starting point is an installed base audit that examines the instrumentation already in place. "Dumb" instrumentation doesn't offer a lot of diagnostic or process information, so it likely won't support a move to this type of predictive maintenance. However, many users may find they have the necessary "smart" instrumentation in place, but have merely been accessing just the basic data. "Most modern instrumentation offers a lot of diagnostic and process monitoring information that is not being taken advantage of, so it may be that processors simply need to take

Seeq

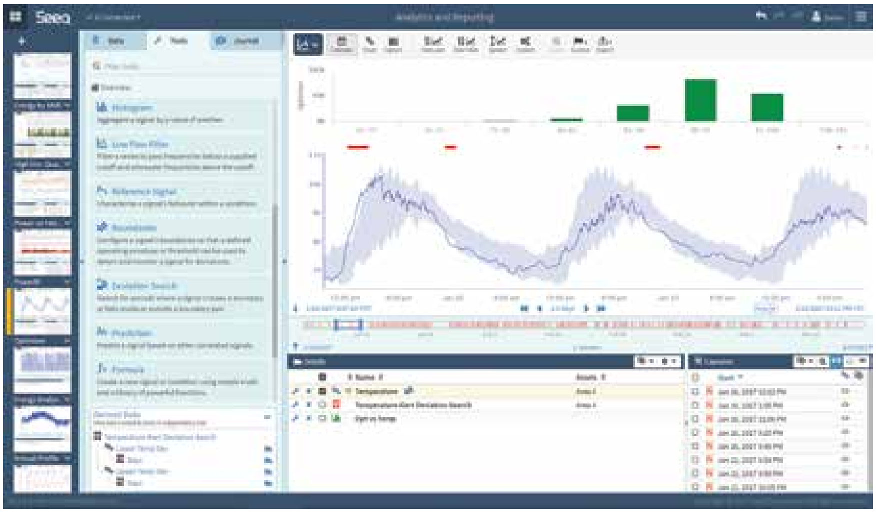


FIGURE 6. Seeq can be used by maintenance professional to calculate ROI of immediate versus deferred maintenance in real time, providing an optimal answer to the question of just when an asset should be repaired

a fresh look at the information that can be extracted from their existing devices. If the instruments don't offer this, it is also possible to strategically add measurement points or different measuring technologies to obtain additional information."

He continues: "We usually tell customers to start with what they have and strategically figure out where it makes the most sense to upgrade. You don't upgrade everything in the plant and completely shift from preventive to predictive maintenance, but rather look at the most important applications, such as those that are critical or involve a lot of expense, and start by implementing a predictive strategy on critical, high-value applications."

Moving data. Once collected, there must be a way to get data out of the instruments to a place where it can be analyzed. Usually a digital communication system takes data to the control room, an edge device that moves data to the historian or the cloud or possibly an interfacing software system where it can be analyzed using some type of analytics methodology. Many automation and software providers offer edge-type devices where analytics may take place (thus the term edge computing) or where data may be streamed to the cloud for cloud-based computation.

"Something to keep in mind when talking about predictive maintenance and analytics in the context of buzzwords like Big Data, Industry 4.0 and the IIoT, is the importance of open standards," says Firas Khalil, head of the Digital Enterprise Lab with Siemens (Alpharetta, Ga.; www.usa.siemens.com/processautomation). "Open standards allow better communication when interfacing with different vendors and suppliers in terms of all the technologies being offered now and in the future. This applies not only to the cloud, but also on the level beyond the cloud, as well as on the device level, IT components, control system and throughout the lifecycle of it all. As the predictive maintenance trend continues to grow, most processors will use a multi-vendor approach, so interoperability will grow increasingly important."

Analyzing the data. "Assessing the information is more than just a black box that receives data and spits out results processors can use to plan maintenance," says Phil Bush, product manager, Remote Monitoring & Analytics, with Rockwell Automation (Milwaukee, Wis.; www.rockwellautomation.com). "It's about translating the data using subject matter expertise and providing process models that can be used to represent normal operations for that process and trigger alerts when something is abnormal, as well as using machine learning to continuously train the system to know what is normal and what will cause an upset."

Getting to this point requires data associated with the process, including historical data because this information will be used for building predictive models. "To predict what will go on in the future, we have to understand what has gone on in the past," says Bush.

Another requirement for being able to build a predictive maintenance program is expertise around the process itself; what is normal and appropriate for the process? "These two aspects depend upon the processors providing the data associated with the process and expertise about their process," Bush says. "Above that, there's usually analytic software and machine-learning technology components that take subject matter expertise and translate it into predictive models that can be used by the processors to make appropriate maintenance decisions."

In addition to software that includes analytics and machine learning for advanced predictive maintenance, some incorporate a "digital twin" that serves as a representation of the asset, complete with dynamic information to provide a complete view, says Hans Thalbauer, senior vice president, Digital Supply Chain and Internet of Things, with SAP (Newtown Square, Pa.; www.sap.com). And, if desired, a network can be created that includes not only information about the asset, its performance and maintenance requirements, but also connects to any partners involved in the lifecycle of that asset, such as third-party maintenance services and other business partners. "These are total solutions that create a complete stack with a digital twin of the asset that includes the asset performance and management strategy, as well as the asset intelligence network, which allows you to connect the maintenance and procurement departments, vendors in the supply chain and anywhere else that might be affected by the asset or tied into the financial element of the asset's performance."

"Integration between all the organizations, components and processes allows users to leverage the information in different ways so the most value can be generated by connecting the machine, analyzing information about the asset and performing necessary actions as part of the business process," says Thalbauer (Figure 5).

The benefits

In today's competitive process environment, predictive maintenance should be used not only to keep the process up and running, but also to provide guidance on how to spend maintenance dollars in the way that provides the greatest return on investment.

For example, some software provides advanced simulation, making it possible to venture beyond just noting that equipment will fail and what that means for the process. "Most simulation will tell you what will happen if a component fails," says Robert Golightly, senior product marketing manager of the asset performance business unit with AspenTech (Bedford, Mass.; www.aspentech.com). "That's sort of a 'binary' decision process, but we make it 'analog' with simulation that looks at the problem on a flow basis and what happens not only if it fails, but when it slows down, runs at full steam or anything in between, which lets users monetize decisions."

For example, it's not unusual for more than one component to require maintenance at any given time, so it often becomes a case of determining which piece of equipment is more important. "Our software can prioritize the information by saying, 'Here's six things that are going to go wrong in the process. These three don't matter, these three do. Work on them in this order.' It is the adult in the room that tells users where to focus based on economic impact."

Even further, if users ensure that all involved costs are considered, including asset value, maintenance costs, analytics infrastructure and uptime of the plant, they can make the right tradeoffs. "For example, run to failure with no maintenance also has a place in the maintenance toolbox, along with preventive and predictive practices, but the costs and tradeoffs must be analyzed for each approach: run to failure, calendar/hours of operation-based preventive and predictive," says Michael Risse, vice president at Seeq (Seattle, Wash.; www.seeq.com). "One customer has created a model with Seeq where they can see declining performance in an asset, and trade off this decrease in asset performance against the market value of the product to decide when to perform maintenance. So rather than optimize for a process parameter or other metric, they are making real-time profitability the priority outcome."

He continues: "That is where leading companies are going, not to the asset or line level, but at the outcome level of increased ROI [return on investment; Figure 6]. This can mean a perfectly good decision is delaying predictive maintenance that is known to be required in order to continue production that will be profitable over the coming weeks or months."

SAP's Thalbauer agrees. "The idea is to build a really intelligent enterprise that includes connectivity and integration between all the components, processes and organizations so that you may leverage the information in multiple ways so you can perform actions as part of the overall business process. In the end, it's about the intelligent connection between the equipment and the business," he says. ■

Joy LePree

Focus on Plant Security

Claroty

Rite-Hite

Larson Electronics

Protect plant assets from cybersecurity threats

The fully integrated Claroty Platform (photo) incorporates realtime vulnerability monitoring and network "hygiene" insights with attack-vector analysis capabilities, allowing industrial asset owners to better protect industrial control systems from cyberattacks. The system includes a comprehensive, integrated suite of products that are designed to provide safe, passive threat monitoring to protect industrial networks. It continuously monitors for new anomalies and vulnerabilities and analyzes pathways to their most important assets. It is suitable for use in electric transmission systems or oil-and-gas pipelines, says the company. The product produces a comprehensive report for plant operators, detailing the industrial network and its assets, to give users deeper insight into the state of the network configuration and any weaknesses. — *Claroty, New York, N.Y.*
www.claroty.com

Dock leveler helps to improve security at loading docks

The RHV-4100 Vertical-Storing Hydraulic Dock Leveler (photo) protects workers from injury and improves efficiency and security at loading docks. With this interior dock configuration, trucks back up to the loading dock with their doors closed. Once they are secured at the dock, the trailer's security seal is broken and the doors are then opened into the buiding. This allows for faster loading and unloading, and helps to increase the security of the facility. The RHV functions as a "power-up/power-down unit," so operation requires constant pressure on the controls. Removing pressure from the "raise" or "lower" buttons immediately stops movement of the leveler. It includes an automatic safety stop that is activated when personnel movement is detected in the leveler pit area during operation. — *Rite-Hite Corp., Milwaukee, Wis.*
www.ritehite.com

Remotely operated camera safeguards plant assets

The EXPCMR-IP-POE-1080P-FIWA explosion-proof security camera provides crisp, clear imagery within the 194-deg horizontal field of view, which makes it ideal for work-site monitoring and general inspections. It provides live feed, with 1,080-pixel resolution, from inside tanks, reactors and other vessels, making it ideal for remote inspection in hazardous locations. The live stream from this camera can be recorded with user-provided equipment. For instance, the camera can be hooked up to network cable and run back to a user-provided DVR system mounted outside the hazardous loation, protecting plant personnel. — *Larson Electronics LLC, Kemp, Tex.*
www.larsonelectronics.com

These courses will strengthen your plant-security expertise

The company's extensive CSP (Cybersecurity Practitioner) Program consists of various IEC 62443-based Industrial Automated Control System cybersecurity courses, which are presented by the company. The courses, related to such topics as safety-critical and high-availability automation systems, control system cybersecurity, alarm management and more, and the overall certification program, are designed to help participants develop and demonstrate competency in assessing and mitigating cybersecurity threats. They are appropriate for participants considering Certified Automation Cybersecurity Specialist (CACS) or Certified Automation Cybersecurity Expert (CACE) certification, says the company. Participants who are able to demonstrate that they have retained the knowledge presented earn a CSP Certificate. — *Exida, Sellersville, Pa.*
www.exida.com

Better signage improves security

The adhesive Surface Safe Sign Labels allow chemical process

Note: For more information, circle the 3-digit number on p. 62, or use the website designation.

Avery Products

plants to improve signage without damaging painted walls, windows or stainless steel, and without leaving a messy residue (photo). The sign labels are made from durable polyester material that is water-, chemical-, abrasion- and tear-resistant. They are used to create caution, hazard, compliance, warning and confined-space signage, as well as signage for office and meeting spaces. They are easy to customize (using the company's template software). Users can print finished labels and signs from a desktop printer, or have them custom-printed by the company. — *Avery Products Corp., Brea, Calif.*

www.avery.com

Adding another layer of protection to control devices

This company has launched Mocana TrustCenter (photo), a new services

Mocana

platform that allows manufacturers and operators of Internet of Things and Industrial Control Systems (ICS/IoT) devices to securely enroll and update them. With TrustCenter, Mocana can automatically manage secure updates of software, hardware and firmware on millions of IoT/ICS devices in seconds, preventing compromises and any malware compromises. This solution aims to not only find vulnerabilities and resolve them with traditional IT security solutions, but also to protect the software and firmware and all components on these devices against cybersecurity threats, using strong authentication and cryptography to verify that the device and software updates are secure. The new services platform complements the company's TrustPoint IoT endpoint security software, which already protects more than 100 million devices today, says the company. The new automated platform can be deployed "on bare metal," or via private cloud or public cloud Infrastructure-as-a-Service (IaaS), and ensures the trustworthiness of both the device and the data from end to end, says the company. — *Mocana Corp., San Francisco, Calif.*

www.mocana.com

Standardizing the approach to managing vulnerabilities

With more than 17,000 cybersecurity vulnerabilities disclosed within the past year, according to this software company, organizations throughout the chemical process industries (CPI) are often forced to rely on a patchwork of security patches, and many process operators operate with relatively scarce internal IT resources. This company recommends a standardized, three-pronged approach to reducing risk and managing chemical process plant vulnerabilities, with the goal of determining risk criticality using vulnerability intelligence, prioritizing

remediation options for known vulnerabilities (based on criticality), and applying patches to mitigate risk with an emphasis on testing in controlled environments. The company's Vulnerability and Software Asset Management (SAM) solutions help process operators to evaluate and procure the right software and cloud services to reduce compliance and security risk. — *Flexera Software, Itasca, Ill.*
www.flexera.com

Integrated components can improve safety and reduce risk

Process Safety Office provides process safety and risk professionals with an integrated suite of tools to support process hazards analysis, auditing, consequence analysis, risk analysis, facility siting and evaluation and design of pressure-relief and flare systems. This versatile toolbox presents a seamless integration of process safety and information technologies for compliance, risk management and business efficiencies to support process-safety-management programs. Each of the components — SuperChems, PHAGlobal, ioViper, ioLogic, ioVu, ioAuditor, ioSecure, User-Defined Component and iViewer — supports different risk-minimization objectives, such as improved consequence analysis related to facility siting, pressure-relief and flare-systems evaluation and design, process hazards analysis, evaluation of vibration-induced fatique in process and relief piping, construction of visual piping isometrics, auditing safety protocols, report generation and more, says the company. — *ioMosaic, Salem, N.H.*
www.iomosaic.com

Cybersecurity solutions safeguard infrastructure

This company's broad technology offerings are designed to protect infrastructure in industries, such as oil-and-gas and power-grid installations. The SNOK Cybersecurity Monitoring Solution monitors both the network and endpoints (hosts) to detect intrusions. It is a scalable, distributed, small-data solution designed to perform local detection of malware, espionage, sabotage or other harmful cyber events, says the manufacturer. SNOK is designed for industrial sites running on hardware with limited computing power. Products in this portfolio include Network Intrusion Detection System (IDS), Endpoint Monitoring, PLC Threat Detection, Enterprise Solution, and Asset Discovery. Customers can request a 30-day trial program and analysis report. — *Secure-NOK, Houston*
www.securenok.com

Use anomaly detection to boost industrial cyber security

Siemens

The Industrial Anomaly Detection solution enables security-related incidents, such as unauthorized intrusions and malware, to be identified and countermeasures to be taken (photo). The software can be pre-installed on an industrial PC and easily integrated into industrial environments, or it can be enabled to run on network components from this manufacturer, such as its multiservice platform Ruggedcom RX1500 with Ruggedcom APE. It is said to be especially well-suited for companies in the chemical, pharmaceutical, food and beverage, water- and wastewater-treatment sectors. The anomaly-detection system incorporates artificial intelligence (AI) with machine learning, to configure the system (for instance, automatically analyzing data traffic in the network in the "learning phase," so it can then reliably detect anomolies that might indicate intrusion or data threat by hackers). If deviations are detected, the system automatically sends an alarm to users. Depending on the criticality, the incidents can be dealt with by onsite experts or external security specialists. — *Siemens AG, Nuremburg, Germany*
www.siemens.com ■

Suzanne Shelley

New Products

Pressure gages now equipped with calibration reminder system

An automatic calibration-reminder system has been added to the XP2i pressure gage (photo). The new system greatly reduces the possibility of using gages after their calibration dates and potentially incurring regulatory fines. Manual record-keeping and notifications are replaced by customizable onscreen alerts prior to the due date, warning alerts on and after the due date, and an optional capability to lock the gage from use after its calibration due date. Dates, reminders and message types are set by supervisors using software. The intrinsically safe XP2i gage offers high-accuracy pressure recording in harsh environments from –10 to 50°C. Key features include an IP-67 rated, marine-grade enclosure, a fast pressure-safety-valve (PSV) mode, custom engineering units and a leak-free pressure fitting connection. — *Crystal Engineering, a unit of Ametek Sensors, Test & Calibration, San Luis Obispo, Calif.*

www.ametekcalibration.com

Digital floor scales with a rugged design

The PowerDeck digital floor scale (photo) combines this company's Powercell digital load-cell technology with a rugged platform design. This new design aims to eliminate the reliability issues typically associated with floor scales and the associated analog load cells, cables and junction boxes, which can lead to weighing errors and disruptions in production. The PowerDeck includes realtime guidance that alerts operators when a product has been improperly loaded and provides instructions for optimal load placement, helping to avoid uneven filling spills and inaccurate batches. The durable platform can handle variable batch sizes and accuracy requirements. By removing the junction box and making the system fully waterproof, maintenance costs are greatly reduced. The system also includes realtime alerts for environmental hazards, such as debris, shock-loading and temperature fluctuations. — *Mettler Toledo, Columbus, Ohio*

www.mt.com

Fully integrated Venturi scrubber units

Series 7000/8000 Venturi scrubbers are now available in a fully integrated package (photo) that includes a recirculation pump, piping networks, instrumentation and automated controls. Units that are designed for outdoor installation are available with a freeze-protection package for operation in cold-weather climates. Series 7000/8000 scrubber packages enable users to meet particulate-emissions requirements and offer low-maintenance operation. They are also available in a wide variety of materials to handle extremely corrosive or erosive gas-contaminant streams. An advanced throat design and diverging section provides high collection efficiencies at reduced pressure drops. Throat sections are available for manual operation or can be equipped with an optional automatic adjustment mechanism for maximum flexibility in meeting the required collection efficiency and gas-volume operating range. — *Bionomic Industries, Inc., Mahwah, N.J.*

www.bionomicind.com

New hose loaders feature a space-saving profile

The Compact Torsion Swivel (CTS) hose loader (photo) features a fully integrated internal torsion spring for a streamlined profile and has been engineered to replace existing FMC TL loaders or any loaders installed in applications where space is limited. The CTS hose loader provides a compact, low-profile solution with simplified maintenance and spring adjustment. The integrated carbon-steel bearing module also helps to greatly increase service life, according to the company. — *OPW Engineered Systems, Inc., Lebanon, Ohio*

www.opw-es.com

These couplings prevent slippage for mixers and more

This company's dual-keyed rigid couplings (photo, p. 28) feature axial and annular keyways to provide additional security for high torque and axial-load applications. The couplings are suitable for heavy, suspended loads on shafts used in mixers, pumps, fans and related drive systems. Designed to prevent axial and radial slippage with higher loads for a given bore size,

Crystal Engineering

Mettler Toledo

Bionomic Industries

OPW Engineered Systems

Stafford Manufacturing

they are available in diameters from 1 to 5 in. with straight-through or stepped bores for accommodating different diameter shafts. The couplings can be machined from high-temperature alloys, steel, stainless steel or aluminum. These rigid couplings are well-suited for connecting supported shafts and for mixers with unsupported shafts and both new and retrofit power-transmission applications. — *Stafford Manufacturing Corp., North Reading, Mass.*
www.staffordmfg.com

This dispersing mixer also has a proprietary planetary design

Charles Ross & Son

The PowerMix Model PDM-10 (photo) combines high-speed dispersion with planetary stirring action, imparting high shear to tough-to-mix applications with low flowability. Beyond a traditional kneading action, the mixing mechanism of this proprietary planetary design is appropriate for heavy pastes and slurry-like media that may include a considerable amount of powders that must be wetted out and dispersed uniformly. These 10-gal models feature an additional sawtooth blade on each disperser shaft, doubling the shear input capacity of the mixer. For more viscous applications prone to climbing up the stirrer, patented High Viscosity (HV) blades are available and offer a helical curvature that pushes product forward and downward. Fully automated recipe controls and data-acquisition systems are also available. — *Charles Ross & Son Co., Hauppauge, N.Y.*
www.mixers.com

A new infrared sensor improves CO_2 monitoring

The Ultima X5000 gas monitor is now being offered with the new XIR Plus infrared (IR) sensor to provide enhanced detection of carbon dioxide (CO_2). The monitor continuously detects CO_2 levels and issues alarm commands when the gas is present in toxic quantities. Two detection ranges for CO_2 gas are available — either 2% or 5% by volume. Highly stable, the XIR Plus sensor features repeatability of ±2% of lower explosive limit (LEL) or volume, and drift is <2% of full scale per year, according to the manufacturer. The sensor's T90 range is less than 2 s, providing fast warning when CO_2 gas is present. The monitor features a touch-

control display, dual sensor inputs and eliminates the need for specialized setup tools or a separate controller. — *MSA Safety Inc., Cranberry Township, Pa.*
www.msasafety.com

New, higher-capacity pump for sand and slurry dewatering

The new KB220 pump (photo) operates at flowrates up to 810 gal/min in sand and slurry dewatering applications. The 30-h.p. addition to the KB Series is available in 460- or 575-V versions and offers a maximum head of up to 186 ft. KB Series pumps are specifically designed and constructed to reliably handle the issues associated with sand and small abrasives encountered in the construction and mining sectors, as well as other applications. The KB220 uses a chrome iron agitator to move settled solids. The pump motor is protected with a double-mechanical seal, which features a separate lip seal, to prevent all leaks. Embedded thermal sensors in the motor windings help to protect the motor from overheating and premature failure. The volute casing is constructed from hardened ductile iron, and durable cables are available for operating in aggressive environments. — *BJM Pumps, LLC, Old Saybrook, Conn.*
www.bjmpumps.com

BJM Pumps

Production crimper with slide-bearing technology

The HM 665 crimping machine (photo) offers an opening stroke of more than 180 mm and an opening diameter of 580 mm (without dies). Using this company's unique slide-bearing technology, the HM 665 can be used to crimp industrial hoses up to 12-in. in diameter with ANSI flanges and pipe fittings up to 16-in. without having to remove the dies. The fixed die and the 250-mm-long bottom die make workpiece positioning easy. Along with hydraulic hoses, the machine can be used to join reinforcing steel, steel cables, ropes and insulators with forces up to 6,000 kN. The HM 665 is controlled through the Control C.2 control unit available from this company. — *Uniflex Hydraulik GmbH, Karben, Germany*
www.uniflex.de ■

Mary Page Bailey

Uniflex Hydraulik

UN Codes for Packaging Dangerous Goods

Department Editor: Scott Jenkins

Shipping and transport of chemicals require specialized packaging to mitigate the hazards that the chemicals may present. Only packages that are certified to have met testing standards issued by the United Nations (UN) Economic and Social Council (New York, N.Y.; www.un.org) can be used to transport dangerous goods, which include most chemicals. The UN has developed a code system for classifying packaging and containers for dangerous materials. This one-page reference explains the UN packaging code designations and provides information on the categories for each.

UN codes

All packages and containers manufactured to be "UN-approved" for holiding potentially dangerous materials are marked with a code that indicates the properties of the container and the physical nature of the product it contains. The UN packaging codes begin with a either a capital "UN" or symbol with a lowercase "u" above a lowercase "n" inside a circle (Figure 1). The codes consist of a series of numbers and letters usually stamped into metal or plastic drums and intermediate bulk containers (IBCs), and are printed on boxes and bags. Packages without the UN certification mark should not be used for dangerous goods.

The "UN" at the beginning of the code states that the package meets the performance standards set forth in the UN Recommendations on the Transport of Dangerous Goods Model Regulations (Orange book) [*1*]. The code's numbers and letters each provide information about categories into which the contained substance falls. Information on what each component of the code means is provided in Table 1 and Refs. 2–4.

The code also includes a year and country, indicating the date and location of the manufacture of the packaging container.

TABLE 1. CLASSIFICATIONS FOR TYPES OF CONTAINERS

Code position	First position (first number after "UN")	Second position (first letter after "UN")	Third position	Fourth position	Fifth position
Definition	Packaging identification code: Identifies the container type	Container material: Identifies the material from which the container is made	Identifies open-head or closed-head containers for drums and multiwall bags or multiwall, water-resistant	Designates which packing group the packaging can be used for (from the hazardous materials table)	Identifies the maximum mass or specific gravity the container has been tested to hold
	Container types	**Container materials**	**Drum type**	**Packing group**	
Code categories	1 = Drum	A = Steel	1: Closed-head drum	X: Packing groups I, II and III	Mass (solids)
	2 = Wooden barrel	B = Aluminum	2: Open head drum	Y: II and III	Specific gravity (liquids)
	3 = Jerrican	C = Natural wood		Z: III only	Hydrostatic pressure
	4 = Box	D = Plywood			
	5 = Bag	F = Reconstituted wood			
	6 = Composite receptable	G = Fiberboard			
	7 = Pressure receptacle	H = Plastic			
		L = Textile			
		M = Paper, multiwall			
		N = Metal other than steel or Al			
		P = Glass, porcelain or stoneware			

Packing groups

Dangerous goods are classified into one of three UN Packing Groups: I, II and III, according to the degree of danger they present. Packing group I represents high danger, while Group II is medium danger and Group III presents low danger.

A key point for shippers with regard to UN packaging ratings is that "overpackaging" is appropriate, but "underpackaging" is not. That is, shippers can use containers that are rated for higher than the properties of the material within, but not lower. For example, for materials in packing Group II, either a X- or Y-rated container is acceptable, but a Z-rated container cannot be used. Packages for Packing Group I will be built to a higher standard and therefore will be more expensive than packages of the same type built for a lower packing group. ■

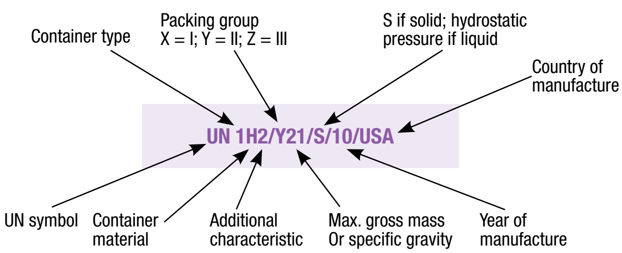


FIGURE 1. Each position in the UN packaging code signifies specific information

References

1. United Nations Economic and Social Council's Committee of Experts on the Transport of Dangerous Goods, UN Model Regulations: UN Recommendations on the Transport of Dangerous Goods, 19th Revision, Parts 4 and 6. United Nations, New York, 2015.
2. New Pig Corp., Cracking the Code: Explaining UN Ratings, New Pig Blog, www.newpig.com, Post accessed June 2018.
3. Tasker, Simon, A guide to chemical packaging, posted to ReAgent Blog (www.reagent.co.uk), February 2012.
4. Label Master, UN Markings Guide, Resources, www.labelmaster.com; accessed June 2018.

Acrylonitrile Production from Propylene

By Intratec Solutions

The highly reactive compound acrylonitrile (AN) contains a carbon-carbon double bond conjugated with a nitrile group. The chemical became important mainly after the 1930s, when it started to be used in the manufacture of synthetic fibers and rubbers.

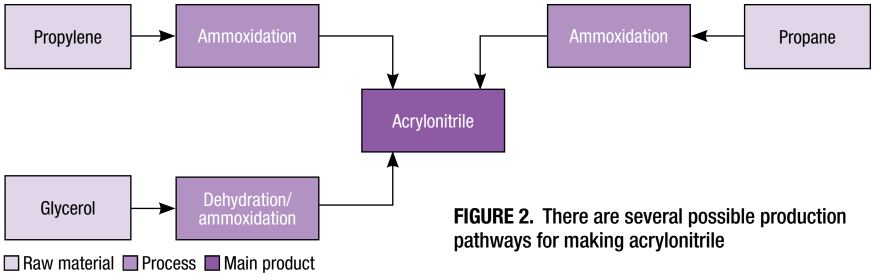


FIGURE 2. There are several possible production pathways for making acrylonitrile

The process

The following describes a process for acrylonitrile production from propylene (Figure 1).

Ammoxidation. Chemical-grade propylene, ammonia and compressed air are fed to a reactor, where an ammoxidation reaction (oxidation of propylene in the presence of ammonia and catalyst) occurs in the vapor phase, over fluidized catalysts. Internal coils remove reaction heat, used for generating steam.

Separation. The reactor effluent is sent to a quench column, where unreacted ammonia is neutralized with sulfuric acid. The cooled effluent gas is partially condensed and transferred to a recovery column. The effluent gases are directed to a nitrile absorber, where acrylonitrile is absorbed in a chilled aqueous stream and transferred to the recovery column.

In the recovery column, acetonitrile and water are separated from acrylonitrile and hydrogen cyanide. While partially purified acrylonitrile product is recovered from the column top, condensed and sent to the first decanter, most of the water is removed from the column bottom and recycled to the quench.

Acetonitrile recovery. A liquid side stream is withdrawn from the recovery column, cooled and returned to the column. Because this stream contains mostly an acetonitrile-water azeotrope, part of it is sent to the acetonitrile stripper, where the acetonitrile-water mixture is distilled under vacuum to separate water and heavy organics, which are recycled to the quench, and a gaseous top draw comprising acetonitrile and water. This acetonitrile-water azeotrope is then distilled at high pressure into three phases: a bottoms product comprising mainly heavy impurities (this is discarded); an overhead stream where water is recovered; and an aqueous stream containing some acetonitrile. This stream is withdrawn from the top of the column and recycled to acetonitrile stripping. Here, high-purity acetonitrile is drawn off as a liquid sidestream.

Purification. The condensed overhead product from the recovery column is fed to a decantation drum and separated into a higher-density phase, which is recycled to the column, and a less-dense phase, which is fed into the HCN column for the separation of HCN from acrylonitrile. The acrylonitrile-rich stream is transferred into the second decanter, in which the denser phase (mainly water) is withdrawn from the decantation drum and recycled to the recovery column. The less dense phase is transferred to acrylonitrile purification, where fiber-grade acrylonitrile product is withdrawn from a side-draw.

Production pathways

Most modern acrylonitrile production is based on vapor-phase ammoxidation of propylene, but there are alternative pathways (Figure 2).

Economic performance

The total operating cost (raw materials, utilities, fixed costs and depreciation costs) estimated to produce acrylonitrile was about $1,800 per ton of acrylonitrile in the second quarter of 2014. The analysis was based on a plant constructed in the U.S. with the capacity to produce 300,000 metric tons per year of acrylonitrile.

This column is based on "Acrylonitrile Production from Propylene," a report published by Intratec. It can be found at: www.intratec.us/analysis/acrylonitrile-e11a. ■

Edited by Scott Jenkins

Editor's note: The content for this column is supplied by Intratec Solutions LLC (Houston; www.intratec.us) and edited by *Chemical Engineering*. The analyses and models presented are prepared on the basis of publicly available and non-confidential information. The content represents the opinions of Intratec only. More information about the methodology for preparing analysis can be found, along with terms of use, at www.intratec.us/che.

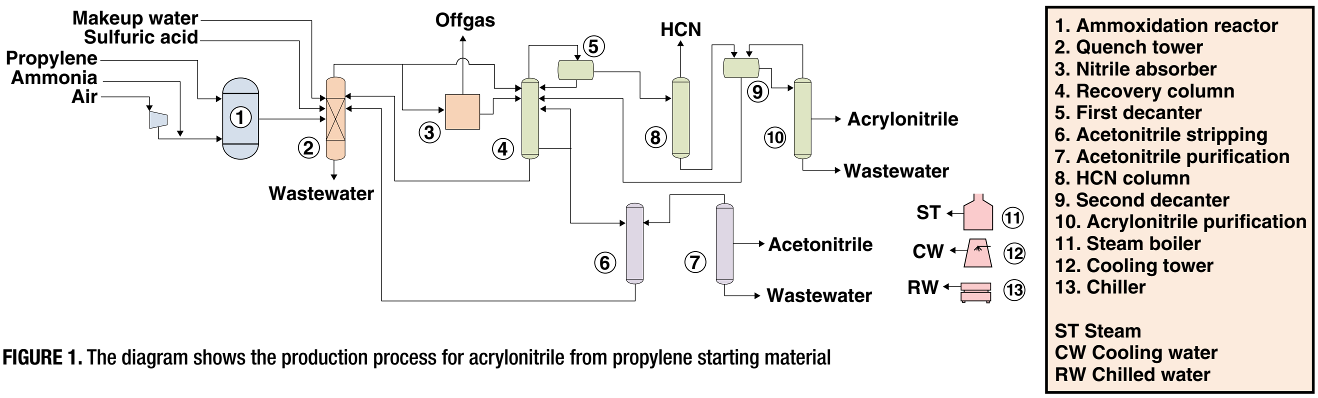


FIGURE 1. The diagram shows the production process for acrylonitrile from propylene starting material

Maintaining Heat-Transfer-Fluid Quality

Learning what can degrade heat-transfer-fluid quality can help minimize potentially negative effects

Conrad Gamble
Eastman

IN BRIEF

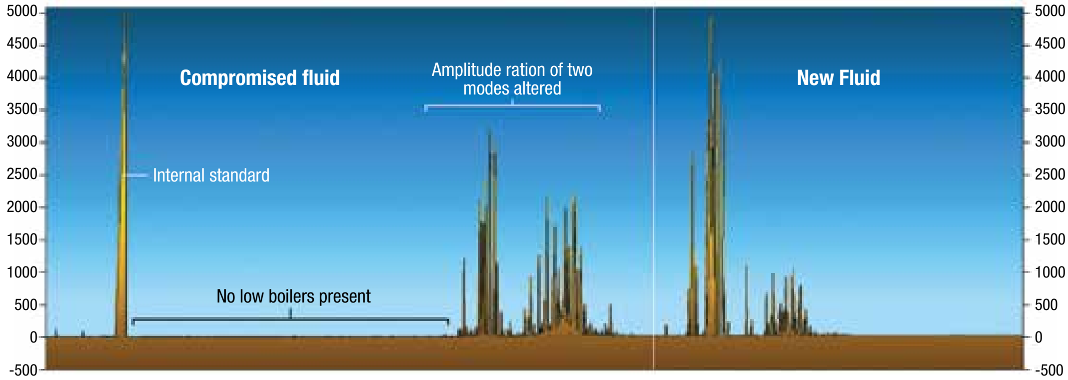


FIGURE 1. On the right, the gas chromatogram of a new heat-transfer fluid with bimodal distribution of peaks is displayed. The left shows fluid after inert gas purging has partially depleted the lower-boiling components (peaks have been shifted to left for illustration). Note that there are no low boilers present to the far left, indicating essentially a complete void of low-boiling degradation products due to the continual gas purge

Organic heat transfer fluids (HTFs) are robust workhorses that are quite stable both thermally and chemically. However, they can yield to attacks from powerful or sometimes even subtle forces, falling short of the hopes and expectations of system owners. The best defense against these attacks is to know the strengths of a fluid and how to strategically intervene. This guide will help educate system owners on how to successfully resist these attacks and minimize impacts of those which may be already underway.

Basic guidelines

Maintaining the fluid quality will minimize its change from like-new condition. In a new system, the first goal is to have the system free of potentially problem-causing debris and residue prior to filling the system with the new heat transfer fluid. It is common for piping and equipment to have a thin rust-inhibitor coating on the metal surfaces that guards them from rusting prior to commissioning. Practical experience has found that small amounts of these coatings have a relatively insignificant impact on the fluid chemistry and stability. As a result, there should be no need for chemical or detergent cleaning and rinsing of the equipment. Of course, any gross contaminants or problem residues, such as from a repurposed vessel, should be locally cleaned.

Preparations before filling. Prior to filling a new system, it should be leak tested — typically with water — to ensure system tightness before filling with the HTF. When draining the water, it is expected that there will be a small amount left behind. Efforts should be made to minimize the residual water and to remove it via warm gas purging or similar methods to greatly facilitate startup. Some use an exit dew point of –30°F (–34°C) as an indicator that the gas-drying step is complete. Excessive water content in the HTF will require a boiling-out process to force the moisture to flash into the vapor phase and be vented from the expansion tank vent. Depending on the system design, this could be a lengthy process. However, it is an essential task to support operation of the fluid at temperatures well above the boiling point of water. Water itself will not react with organic heat transfer fluids within their intended operating temperature ranges. Drying of the excess water from a system leaves the heat transfer fluid with low parts-per-million concentrations of water and undamaged from

the experience. Alternatives to hydrostatic testing with water include pressurized gas with soap solution and low-pressure helium leak detection.

Preventing oxygen exposure. With the fluid heated to temperatures above 175°F (80°C), an organic fluid should be protected against exposure to oxygen in air. This can be accomplished by providing an inert gas blanket within a closed expansion tank or ensuring the fluid temperature within the expansion tank remains cool enough to resist oxidation. The proper design for inerting is to establish a static blanket of positive internal pressure. This keeps oxygen out of the system and protects the fluid from oxidation [*1*]. If a continual purge or sweep of gas is provided, this can gradually deplete the heat transfer fluid of its lower-boiling components, shift the average molecular weight of the fluid higher, and lead to increasing viscosity. The fluid composition and performance can change for the worse, deviating from like-new condition — sometimes to the point that a partial replacement of fluid will be needed to adequately restore its performance. The change from new to compromised fluid composition from this mechanism is illustrated in Figure 1.

A secondary route of vulnerability to oxidation is when heated fluid is drained from the system. Even if collected in clean and dry vessels, oxidized fluid should not be returned into the system. A best practice to permit continued use of drained fluid is to establish job plans that allow the fluid to be cooled to less than 175°F (80°C) prior to draining into clean vessels. Cooling to below 140°F (60°C) will further protect personnel from potential thermal burns in case of skin contact.

Thermal stresses. Next, the potential for excessive thermal stressing of the fluid should be considered. The first consideration of fluid selection is to properly match the fluid to the requisite temperature requirements. A fluid operating above its thermal stability limitation will be damaged by the routine requirements of the process. A properly rated fluid should have the ability to continuously operate at the required supply temperature to the process heat users while providing long and acceptable life [*2*]. In the real world, cir-

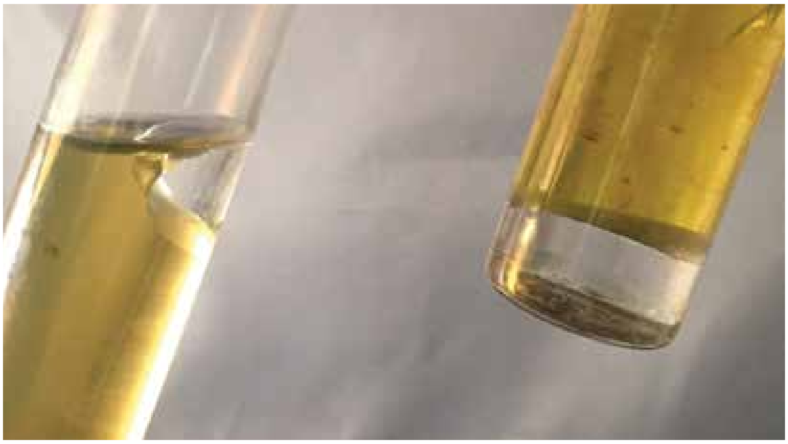


FIGURE 2. Fluids of differing density and solubility can result in localized issues based on position of the potentially troublesome layer

cumstances sometimes create unplanned high-temperature excursions beyond the maximum temperature rating of the fluid. If the maximum bulk-temperature rating of the fluid exceeds the heat-transfer-fluid supply temperature requirements, the fluid has built-in capacity to withstand some short-duration and limited temperature spikes or excursions. Causes of these temperature spikes can include heater-firing controls overshoot, flow imbalance within the heater coils, and power failures that interrupt fluid flow through the heater. Should these circumstances exist, efforts should be made to identify their causes and implement corrective measures before the accumulating impacts of these events irreparably damage the fluid. Some solid-fuel-fired heaters may be more vulnerable to temperature spikes due to the nature of the fuel; hence, a fluid with excess thermal stability capacity is expected to be more resistant to degradation.

FIGURE 3. Acidic corrosion can lead to steel thinning and perforation

Typically, low-temperature extremes should not damage an organic heat transfer fluid. Fluids composed of multiple components will not transition into a crystalline solid form, but will experience increasingly higher viscosity as the temperature decreases. Some fluids with a small number of components, such as eutectic biphenyl and diphenyl ether blend, will freeze into a solid form. On warming, the organic fluid chemistries return undamaged to their liquid state. Organic fluids that do not freeze into crystalline solids should also pose no risk of freeze damage to piping or tubing, which is common with water ice.

Contamination. A potentially more immediate chemistry change of a fluid can result from in-leakage from a process-side stream of greater pressure. This ingress is immediately apparent if the process stream is aqueous and at temperatures above 100°C, since the water can rapidly vaporize and expand to hundreds of times its liquid-phase volume. Smaller leak rates of aqueous streams can be diagnosed by the onset of pump cavitation, increased system pressures, and audible gurgling sounds inside the piping and expansion tank. While the water itself should not chemically interact with the heat transfer fluid, the other process stream components may create issues, such as insoluble solids or flash-point depression, or by introducing thermally unstable organics that quickly break down and either foul heat transfer surfaces or deposit coke within heater coils. Consideration should be given to potential system impacts if such process contamination occurs, with heat-transfer-fluid selection criteria evaluating the ability to best manage the contamination with the least negative consequences. Figure 2 illustrates a mixture of relatively immiscible fluids of differing densities.

Mixing fluids. Just as process contamination can be accidental, the accidental addition of the wrong make-up fluid can happen as well. Actual examples of this unplanned mixing include the addition of drums of glue and JP-8 jet fuel into a heat-transfer-fluid system. Typically, other organic chemistries are of lower thermal stability and can break down quickly. If they break down into lower-boiling compounds, it may be possible to vent to a safe location from the system expansion tank. Higher-boiling compounds formed will often contribute to sludge formation. Some compounds can rapidly form coke

deposits in the highest temperature zone of the fluid within the heater coils. If this occurs, the cooling action of the heat transfer fluid is greatly reduced and can cause the coil wall temperature to increase, leading to blistering or rupture. Consequences can be rapid and severe, so action in response to process contamination and improper make-up fluid addition should be prompt.

Another example of improper fluid addition into a heat-transfer-fluid system is the return of condensate collected from the system vent stream. While this condensate was once within the system itself, it may have chemically changed due to oxidation within the vent condensate collection tank. Return of the fluid into the system will introduce organic acids into the system. These acids can increase the risk of corrosion to carbon-steel components and increase the viscosity of the fluid, which can reduce its heat transfer efficiency. Of course, if water was present in the returned condensate stream, the evolution of water vapor within the system will be immediately apparent. If the condensate collection tank is shared among multiple processes, the stream can also contain contaminants from other processes.

Consequences

What are the consequences of heat-transfer-fluid quality changes? With increases of acidity from either process contaminants or fluid oxidation, the carbon-steel components of the system are at increased risk of corrosion. Most commonly, this is first observed in the vapor space region of the expansion tank where topside nozzle welds can develop pinholes. If the expansion tank is open to the atmosphere and its contents are below 100°C, some organic fluids with lower density than water will allow the water and acids to collect in the bottom of the tank, leading to vessel bottom corrosion (see Figure 3). As previously noted, organic contaminant degradation products, as well as those of significantly degraded heat transfer fluid, can lead to the precipitation of insoluble solids and the formation of deposits onto heat exchange surfaces. These conditions can increase fouling resistance to heat transfer and reduce the thermal efficiency of the system [*3*]. In severe instances, the solids can obstruct instrument sensing lines and increase risk of premature pump-seal failures if solids lodge in-between seal faces. Many times, oxidized fluid experiences a significant viscosity increase, reducing its heat transfer efficiency and increasing energy usage. If the heater-outlet-temperature set point is adjusted upward to compensate for the reduced efficiency, increases to fuel usage, costs, and the thermal degradation rate of the heat-transfer-fluid result.

KEYS TO MAINTAINING HEAT-TRANSFER-FLUID QUALITY

1. Match heat transfer fluid to the process temperature demands.
2. Avoid problem-causing contaminants.
3. Protect against heat-transfer-fluid oxidation and excessive purging and venting.
4. Identify and correct causes of high-temperature excursions.
5. Establish a working relationship with heat-transfer-fluid supplier technical resources.
6. Establish and maintain appropriate administrative and engineering controls.

Some foreign contaminants can have such low thermal stability that they quickly form coke deposits within heater coils, leading to coil overheating, blistering and possible rupture. Monitoring of heater performance to detect the onset of such consequences can include heat-transfer-fluid temperature rise, pressure drop, and flowrates through each coil pass of the heater. With properly balanced flowrates, these measured parameters should be common to each pass. Monitoring of heater stack-gas temperatures, fuel usage rates, and coil skin temperatures using infrared imaging can also be helpful.

Some HTF chemistries can exhibit increased thermal degradation rates when affected by elevated concentrations of impurities or contaminants that can act as free radicals to promote molecular changes [*4*]. As a result, the usable life of the heat transfer fluid is diminished. When production units cannot afford to shut down for a proper identification and repair of leaking equipment and replacement of the fluid, some have chosen to perform partial fluid replacements online. This is a costly approach to extend the operation until a planned downtime, and the subsequent total fluid replacement remains necessary. Contaminated fluid may have additional disposal issues to manage as well, such as one user whose process inadvertently partially halogenated the heat transfer fluid due to in-leakage. The named impacts to operations all act toward increased frequency of unplanned downtimes and increased maintenance costs.

Corrective actions

What if the heat transfer fluid has already fallen victim to one or more of the problems mentioned? The priority is to correct the root cause(s) that led to the deteriorated fluid quality, whether it be equipment failure, human error or other issues. The fluid quality should be assessed. This typically involves the fluid experts consulting with the heating system owner's experts to determine if the fluid quality requires improvement. Analysis of a representative sample of the suspect fluid should be conducted. Fluid quality adjustments can involve a partial to full fluid replacement. This will dilute any contaminants, degradation products, insoluble solids, moisture, and acidity proportional to the fraction of fluid replaced. Added attention to fluid filtration can be an appropriate measure for further insoluble solids removal.

Severe situations where fouling and deposits of solids have occurred can require emptying the system for a more thorough cleaning of the equipment and piping. Options can include solvent cleaning, organic fluid flushing, or water-based chemical cleaning [*5*]. Consultation with the heat-transfer-fluid supplier can be helpful in determining the approach best suited for restoring the system cleanliness, while minimizing costs of cleaning and fluid disposal and keeping the turnaround time to a minimum.

If the heater coils may have been compromised, one useful technique is thermal scanning. While limited to designs permitting the view of an imaging camera through an inspection port for visibility of coils near the combustion zone, this approach can be used to trend coil skin temperatures over time to assess the potential ongoing accumulation of internal coke fouling. The coke can act as an insulation layer, resisting heat transfer into the fluid. As a result, the coil wall or skin temperature increases compared to normal. With diligence in periodically measuring coil skin temperatures, it is possible to have early warning that can prompt corrective measures prior to coil failure. It is important to realize that without baseline temperature measurements when coils are free of coke, measurements taken later with coke deposits present cannot be judged as "normal" or "elevated."

If the heat transfer fluid is being excessively thermally stressed due to process temperature requirements being beyond the capability of the fluid to withstand, alternate fluid chemistries should be considered. Consultation with the heat-transfer-fluid supplier can usually identify at least one or two viable alternative fluids of greater thermal stability to consider. With any upgrade of fluid chemistries, a variety of engineering evaluations and actions should be undertaken, including potential changes in fluid volume expansion (expansion tank sizing), materials of compat-

ibility, pressure-relief-device sizing, heater design calculations, pump sizing, instrumentation calibrations, hazard communication training for personnel, and more.

In the follow-up to any event that compromised the heat transfer fluid, corrective actions should be implemented to prevent recurrence. These can be in the form of administrative controls, such as refresher training, revised procedures, new process signage, or updated policies focused on intervening in the identified causes. They may also include engineering controls, such as modified control system logic, equipment changes, operating condition changes, or changes of the heat transfer fluid itself.

Final thoughts

Perhaps the best approach to managing fluid quality is prevention of the common problem causes. This starts with a good system design that can manage the effective venting of moisture and low-boiling thermal degradation products from the system. It also requires knowledge of the optimal expansion-tank design features. Any proposed heat-transfer-fluid system design or modification should be reviewed by an expert for comments and insights on the ability of the design to support fluid maintenance.

Next, it is important to establish a good fluid sample-analysis monitoring program, beginning with a baseline sample in the first month of operation. Samples should always be collected from the same sample port for consistency and from a location where the fluid is well mixed. Ask the fluid supplier for the recommended sample frequency, but annual sample analysis is most common for routine monitoring. Intermediate samples should be considered if process upsets are suspected to be fluid related.

Even a properly designed and constructed system can have problems if operated and maintained inadequately. Ensure the personnel responsible for the operation of the system are knowledgeable and are provided with adequate training to avoid or minimize excursions that can impart stresses to the fluid and system components. When concerns are raised, they should be promptly investigated so that corrective actions can be implemented before problems become undesirably large or costly to address.

Lastly, system owners should establish a good relationship with their fluid supplier's representatives. These experts have vested interest in supporting their products' users to maintain their satisfaction, to optimize the performance and life expectancy of the heat transfer fluid, and to bring technical specialist resources to assist their customers quickly and effectively — usually at little to no added cost. The user of the heat transfer fluid should utilize this important expert resource, which is at their disposal, so that their focus can remain on their own production metrics. ■

Edited by Dorothy Lozowski

References

1. Technical Information Bulletin No. 4, Heat Transfer System Expansion Tank Design, Eastman, p. 3.
2. Liquid Phase Systems Design Guide, Eastman, p. 4.
3. Therminol® Information Bulletin No. 2, In-Use Testing Of Therminol Heat Transfer Fluids, Eastman, p. 3.
4. Gamble, C. and Schopf, M., "Optimization Opportunities for Therminol® VP-1 Heat Transfer Fluid in Concentrating Solar Power Facilities," Solutia Inc., 2009.
5. Therminol® Information Bulletin No. 1, Cleaning Organic Heat Transfer Fluid Systems, Eastman, p. 3.

Author

Conrad Gamble, P.E., is the product steward for Therminol heat transfer fluids at Solutia Inc., a subsidiary of Eastman Chemical Company (Anniston, AL; Phone: 256-231-8525; Email: cegamb@eastman.com). Gamble joined Solutia Inc. (then Monsanto Co.) in 1985 as a process engineer. He served for 11 years in heat-transfer-fluid process engineering, manufacturing and engineering roles until his current assignment as senior technical specialist. Since 1999, he has supported the Therminol heat-transfer-fluid business in product development and customer technical service and provides customer educational training. A licensed professional engineer and recognized expert in the industry, Gamble has served the chemical industry for more than 30 years. He is also a member of the American Institute of Chemical Engineers. Gamble holds a B.S.Ch.E. from the University of Alabama in Tuscaloosa.

Part 1

An Overview of Gas Flow Measurement

Many applications require accurate flowrate measurement for air or other gases. Installation requirements, calibration and maintenance are among the factors that must be considered when selecting a flow-measurement technology

Art Womack
Fluid Components International (FCI)

IN BRIEF

- COMMON MEASUREMENT APPLICATIONS
- GAS-FLOW CHALLENGES
- GAS-MEASUREMENT TECHNOLOGIES
- FLOWMETER CALIBRATION
- INSTALLATION CONSIDERATIONS
- MAINTENANCE REQUIREMENTS
- ACCOUNTING FOR COST

The measurement of air and other gases in manufacturing plants in the chemical process industries (CPI) is essential in many functional applications, including safety, process control, product quality, production efficiency, environmental compliance and costs. When the measurement of air or other gases is inaccurate or inconsistent, some potential outcomes include serious accidents, emergency shutdowns, unplanned maintenance, production slowdowns or cost overruns.

There are six to eight viable flow measurement technologies for gases available today, but only about half of them are suitable for the heavy-duty metering applications found in the most challenging CPI operations. Each technology has its own strengths and weaknesses, depending on exactly what material needs to be measured, the required accuracy, where it needs to be measured and so on.

The truism "knowledge is power" definitely applies when it comes to choosing a flowmeter for gas measurement tasks in CPI plants. The same flow-sensing technology that is chosen for one application in a plant is quite possibly the wrong choice in a different application — even one that is in close proximity within a facility.

The cost of choosing the wrong flowmeter — in terms of extra maintenance, repairs and spares in large CPI plants — can add up quickly to tens of thousands of dollars. If safety events, poor product quality, a production slowdown or environmental compliance issues occur, then the cost of failing to recognize the subtle differences in air and other gas flow-measurement technologies can be punitive.

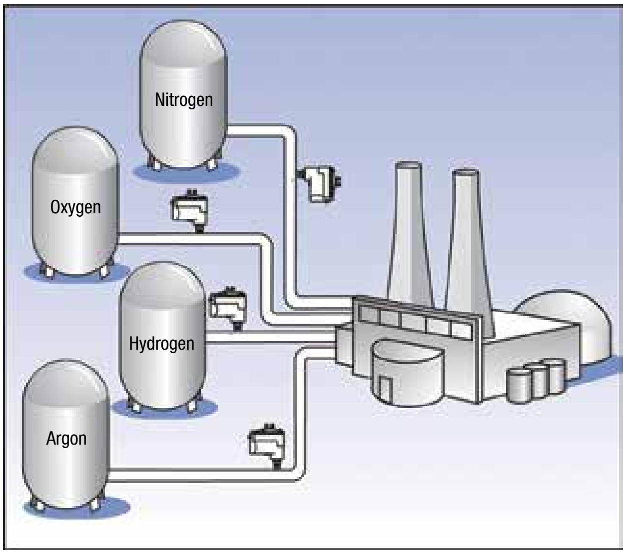


FIGURE 1. Gas distribution metering requires accurate flow measurement because diverse applications may require varying volumes of gas

Common measurement applications

Flowmeters are used to measure the flowrate of air or other gases, as well as totalized flow. Due to the hazardous operating environments that may be encountered in CPI plants, gas flowmeters generally require approvals for hazardous areas (for instance, HazEx) and often must be compliant with the IEC 61508/61511 (SIL) standard as part of a Safety Instrumented System (SIS) in many applications. Four of the most common and the most demanding gas flow-measurement applications in chemical plants are described in the following sections.

Gas distribution metering. Many chemical processes require large varying volumes of specific gases, such as nitrogen, argon and oxygen for inert ions or purging or blanketing. Hydrogen may be required as

a catalyst and other specific gases are used as well (Figure 1). The accurate measurement of these gases is necessary for process control, gas-inventory control and cost management.

Flaring systems. In petrochemical production, refining and storage, flare gas systems are used to burn off and dispose of waste, excess or off-gases and as a safety system (Figure 2). The accurate, responsive and reliable measurement of flare gas is essential in order to assure proper operation of the flare gas system, which protects people and equipment from hazardous combustible gases to maintain a safe working environment and to avoid environmental contamination.

Tank blanketing. Nitrogen blanketing is used in the chemical and petroleum refining industries to reduce the hazards associated with flammable liquids, which helps to support plant safety and can also increase productivity. Blanketing or padding is the process of applying inert nitrogen gas to the vapor space of a tank or vessel (Figure 3), which minimizes the possibility of an explosion or fire by reducing the oxygen content or the concentration of flammable or explosive vapors.

Stack gas monitoring. Measuring the output of plant waste gases through large stacks with scrubber systems requires multiple flow sensors, which are placed in strategic locations (Figure 4). These stack-gas systems are critical for ensuring environmental compliance. Stack continuous emission monitoring systems (CEMS) must meet several standards, including: U.S. Environmental Protection Agency (EPA) 10 CFR 40 and 40 CFR 98; E.U. Directives 2003/87/EC and 2007/589/EC; U.S. MMR 30 CFR Part 250, Subpart K, Section 250; and others.

Gas-flow challenges

Applications that demand accurate, dependable gas-flow measurement present challenges to process and instrument engineers. The following sections describe specific considerations that require careful attention when choosing a flow measurement or sensing technology.

Low and high flows. Sensitivity to low-flow conditions is required to identify and measure leaking valves and the normal low-flow operation associated with day-to-day operations. The capability to measure very high flows is needed during system upset conditions, requiring a meter that needs to measure flow accurately over an extremely wide turndown range.

Meter calibration. It is essential that flowmeters be calibrated specifically for hydrocarbon composition gases and to match actual process conditions.

Large line sizes. As pipe sizes increase, the number of effective and suitable flowmeter sensing technologies decreases. It becomes imperative that special considerations are

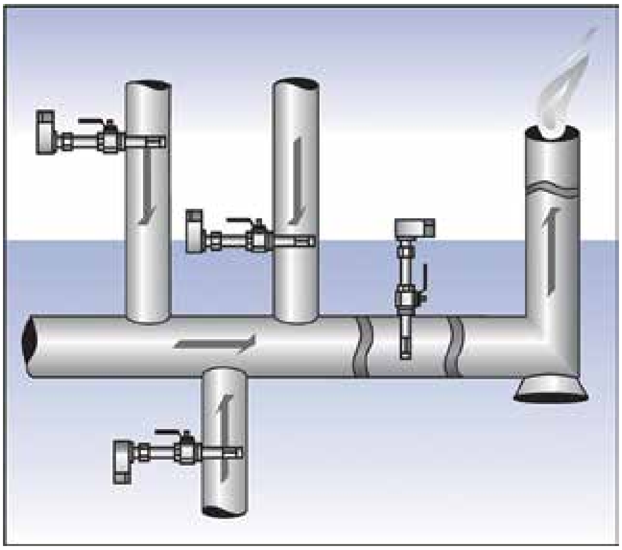


FIGURE 2. Measurement of flare gases is crucial to ensure that systems are operating safely and reliably

taken when selecting a flowmeter for larger lines.

Available straight-run. All velocity-based flowmeter technologies require a certain amount of straight-run pipe both upstream and downstream of the meter in order to achieve accurate flow measurement. These straight-run requirements may not be achievable in crowded production sites and process plants.

Limited access. Access and re-access to piping for installation, maintenance or servicing is frequently difficult. For example, spool-piece flowmeters can require prolonged process shutdowns and extensive onsite labor costs to install and continuously maintain the system, as opposed to insertion-style meters that can be easily inserted into or retracted out of the process through a ball valve.

Agency approvals. When installing meters in hazardous (Ex) locations, the entire flow-metering instrument should carry agency approval credentials for installation in environments with potential hazardous gases. Note that enclosure-only ratings are inadequate.

Gas-measurement technologies

There are two basic types of flowmeters: liquid and gas. Liquid is primarily measured in terms of volumetric flowrate, while gas is a mass-flow measurement because of the unique properties of gases when compared to liquids. While some volumetric technologies can measure gas flowrates, there can be problems with totalized flow. Generally, the best choice is mass-flow sensing technology when measuring air or other gases — especially in critical applications.

Coriolis. The principle of operation for Coriolis flowmeters relies on a vibrating tube where the flow of fluid causes changes in frequency, phase shift or amplitude, which is proportional to the mass flowrate. Coriolis meters are highly accurate and are frequently used in custody transfer applications, but they are on the expensive side and require labor-intensive inline applications.

Differential pressure. Differential pressure (DP) meters and sensors come in several designs, including orifice plates, pitot tubes and Venturis. The typical DP meter designs require the fluid to move through or past two points of reference, creating a differential pressure rate that is equivalent to the rate of flow using the Bernoulli equation with some modifications. If the gas is dirty, there can be orifice clogging issues that require frequent maintenance in order to maintain accuracy.

Ultrasonic. Flowmeters designed with ultrasonic flow-sensing technology rely on ultrasound and the Doppler effect to measure volumetric flowrate. In ultrasonic flowmeters, a transducer emits a beam of ultrasound to a receiving transducer. The transmitted frequency of the beam is altered linearly by particles or bubbles in the fluid stream. The shift in frequencies between the transmitter and receiver can be used to generate a signal proportional to the flowrate.

Optical. Flowmeters designed with optical sensing rely on laser technology and photo detectors. This technology requires the presence of particles in the gas stream. These particles scatter the light beam, and the time it takes for these particles to travel from one laser beam to the other laser beam can be used to

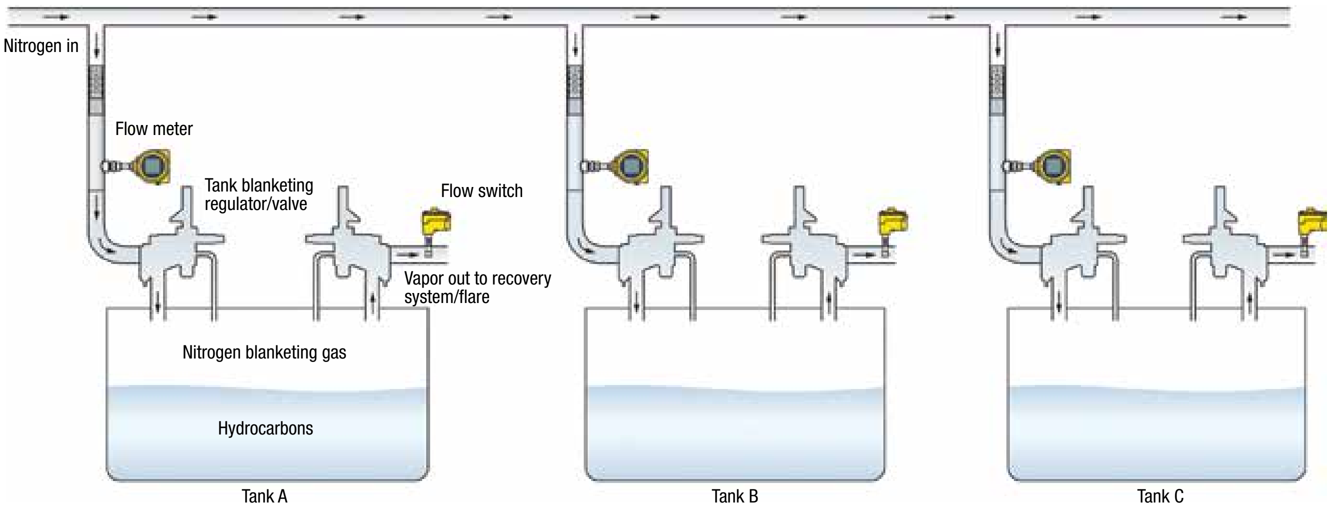


FIGURE 3. Tank blanketing involves filling the inert space of a tank or vessel with an inert gas (typically nitrogen) to reduce fire risks

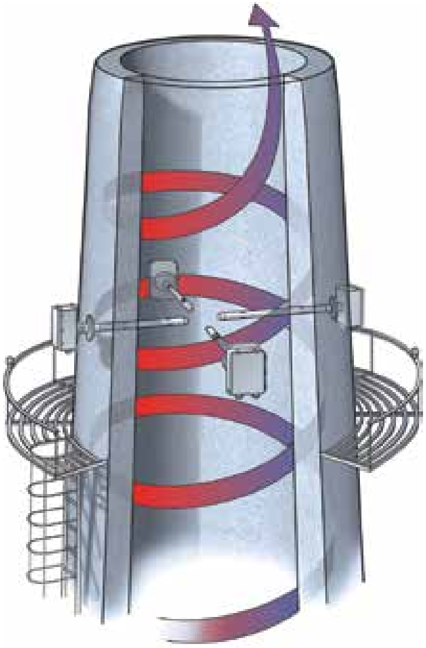


FIGURE 4. Stack-gas systems usually involve a scrubber unit where the gases are monitored using several flow sensors placed in specific locations on the unit

FIGURE 5. Understanding meter calibration is essential for engineers. Instrument manufacturers may offer direct calibration in a dedicated laboratory with traceable equipment and methods

calculate the gas velocity and volumetric flowrate. These meters have good accuracy and wide turndown, but are traditionally expensive.

Thermal dispersion. Flowmeters with thermal-dispersion sensors provide direct mass-flow measurement. Two thermowell-protected platinum resistance temperature detector (RTD) sensors are placed in the process stream. One RTD is heated while the other senses the actual process temperature. The temperature difference between these sensors generates a voltage output, which is proportional to the media cooling effect. This information can be used to measure the gas mass flowrate without the need for additional pressure or temperature transmitters.

Flowmeter calibration

In measuring flow accurately, second only to selecting the proper flow sensing technique is the method of calibration. There are two methods used in calibrating gas flowmeters, as follows:

1. The direct method, where the meter is calibrated to a specific pure process gas or to the actual components of a mixed gas in use
2. The air equivalency method, where the meter is calibrated using air, and then the calibration is adjusted with a pre-defined correction factor

It is important to ask your supplier about the method of flowmeter calibration. Users should know if manufacturers contract out for calibration and if so, with whom, or if they operate their own calibration laboratory (Figure 5) with direct-method calibration test stands and equipment that is traceable in accordance with NIST and ISO/IEC 17025 standards (Figure 6).

Installation considerations

When choosing a flowmeter technology for air or other gases, one of the most important criteria to consider is the location and the manufacturer's installation requirements. Most flowmeter technologies require a stable fluid-flow profile upstream and downstream from the point of meter installation — this is usually defined as equivalent to a specific number of

FIGURE 6. Calibration test equipment should be traceable in accordance with various industry standards

FIGURE 7. Inline (right) and insertion-style (left) flowmeters require different installation considerations, and one method may be preferred depending on specific process needs

pipe diameters of straight-run pipe in each direction. Flow sensors are potentially sensitive to swirling air or gas conditions in the pipe, pressure drops (turndowns) or flow surges. In many cases, irregular flow issues can be solved with flow conditioners. There are various types of flow conditioners that can be inserted strategically in the pipe to "straighten" the flow before it reaches the flow sensor. They may consist of tabs, honeycombs, vanes or other designs, which all serve to straighten the flow. Some straighteners, such as the tab type, actually speed up the rate of flow by creating regular vortices to avoid any loss of gas throughput (pressure drop).

There are two ways to install a flowmeter: inline or insertion (Figure 7). Inline flowmeters must be installed horizontally inside a section of the pipe. Insertion flowmeters are top-mounted through a tap point.

Some flowmeters can only be installed using one method. Venturi meters, for example, must be installed inline (inside the pipe). In comparison, thermal meters, some DP meters (orifice plates) and others can be installed in either inline or insertion configurations.

Lastly, when considering installation requirements, some flowmeter technologies rely on direct mass-flow sensors. Other flowmeters infer mass flow and require pressure or temperature sensors to be installed nearby, along with transmitters, or multivariable transmitters, which can add to their cost and installation complexity.

Maintenance requirements

All flowmeters require maintenance. Some technologies, however, may require more maintenance than others. The type of fluid to be measured can have a major impact on maintenance needs. Pure process gases in a benign plant environment are generally going to have less impact on a flowmeter than dirty waste gases.

Some meter designs require less cleaning or are easier to clean than others. For example, top-mount insertion-style meters with packing glands can be quickly pulled out of the pipe without shutting down the process and cleaned in place with compressed air and subsequently returned to service.

Accounting for cost

There are many factors to consider when choosing a flowmeter for CPI applications. A thorough checklist of considerations would include the following:

- Accuracy
- Repeatability
- Flow sensor technology
- Calibration type
- Installation requirements
- Maintenance
- Cost

In considering the cost of a flowmeter, there are three crucial factors to think about: the purchase price of the meter; the installed cost; and the lifecycle cost.

Stopping your analysis at the purchase price is misleading when it comes to reviewing the true cost of instrumentation — this is especially true for flowmeters.

The two previously discussed options for flowmeter installation — inline and insertion — may result in large differences when it comes to costs. Flowmeters installed in an insertion configuration are simpler to install, which is going to generally result in a lower installed cost versus a flowmeter that is less expensive to purchase, although it requires inline installation.

The last factor to consider is the lifecycle cost. How long does the manufacturer expect the flowmeter to remain in service? Is its life span 5, 10 or 20 years? Over that lifetime, what kind of maintenance will be required? Some meters have movable parts that can break and require repair. Some meters depend on small orifices that tend to narrow or clog in dirty environments, requiring cleaning. These expenses can add up over time, which increases the cost of ownership.

In conclusion, knowledge and experience with flowmeters is power. The more that engineers know about flowmeter technologies, the more thorough and effective the selection process will become. ■

Edited by Mary Page Bailey

Author

Art Womack is a senior applications engineer at Fluid Components International (FCI; 1755 La Costa Meadows Drive, San Marcos, CA, 92078; Phone: 760-744-6950; Email: awomack@fluidcomponents.com.) He has over 20 years of experience in the design, production, application, sales and marketing of flow, level, pressure and temperature process instrumentation. He holds a B.S. in electrical engineering from Rose-Hulman Institute of Technology in Terre Haute, Ind. Prior to working at FCI, Womack held positions at SOR Controls Group and Dwyer Instruments.

Part 2

Measuring and Controlling Flow of Dangerous Materials

Processes that handle extremely dangerous chemicals require robust instrumentation. This article draws on experiences in a phosgene-handling plant to provide guidelines for selecting, installing and maintaining flowmeters safely

Daniel Siddiqui and Nathan Hedrick
Endress + Hauser

IN BRIEF

Phosgene ($COCl_2$) is a chemical used to make plastics and pesticides. A small exposure to just 400 parts per billion is enough to kill a person within 4 to 10 hours. If not detected early, then it can already be too late to save a person's life. Any company dealing with phosgene has to comply with many safety precautions, standards, procedures and so on. Complicating the matter further, phosgene is corrosive, and additional steps must be taken to protect equipment.

One may ask why phosgene is produced if it is so deadly, but phosgene is a required component in a number of industrial products, so its continued use is a necessity. Phosgene is an industrial reagent and building block in the synthesis of pharmaceuticals and other organic compounds. The great majority of phosgene is used in the production of isocyanates, the most important being toluene diisocyanate (TDI) and methylene diphenyl diisocyanate (MDI). These two isocyanates are precursors to polyurethanes. Phosgene is also used in thermoplastics and coatings, such as hexamethylene diisocyanate (HDI). The end uses for these products include flexible polyurethane foams and other polyurethane-based products.

This article discusses the problems involved in the measurement and control of crude TDI. In TDI manufacturing processes, unreacted phosgene and *ortho*-dichlorobenzene (ODB) solvent are encountered in a single process unit, making safe handling and reliable control essential priorities. Included in the article are lessons learned that are applicable to many plants and processes using phosgene and other hot, corrosive or poisonous chemicals. The guidelines provided in this article may also be used in applications that handle other hazardous chemicals, such as MDI, HDI and certain thermoplastics, in many sectors of the chemical process industries (CPI), including plastics, coatings, polycarbonates and polyurethane.

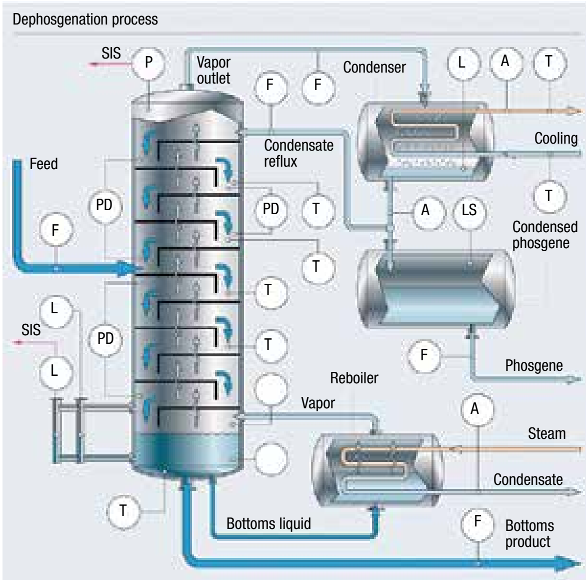


FIGURE 1. Processes that handle poisonous chemicals, such as phosgene, require reliable instrumentation to eliminate the risk of leakage

Dealing with dangerous materials

Because of the dangers, special handling and equipment are needed when working with phosgene. Major phosgene producers follow several guidelines and requirements for process safety, including:

- Emergency response planning based upon best practices defined by producers and the American Institute of Chemical Engineers (AIChE; New York, N.Y.; www.aiche.org)
- The U.S. Occupational Safety and Health Administration's (OSHA; Washington, D.C.; www.osha.gov) Process Safety Management rule (29 CFR 1910.119), which requires process hazard analyses (PHAs) to be conducted every five years where the standard is applicable, and periodic audits are required at least every three years under process safety regulations from OSHA
- Management regulation (29 CFR 1910.119(o)), which ensures that personnel are following the standards and utilizing their training

Special materials of construction are required for piping, equipment and instrumentation, such as tantalum, Hastelloy C, Monel, Inconel and so on. Piping, valves and other components require specialized testing, while pumps and instruments must have special designs and seals. As previously described, phosgene is very poisonous, so even small gas pockets in serviced equipment may be dangerous to maintenance personnel.

Fully enclosed containment is required for newer phosgene facilities, mitigation systems, such as scrubbers, are required, and safety shutdown systems are needed to take the process to a safe state under abnormal conditions. A phosgene monitor or analyzer must be employed to monitor for leakage.

Making TDI

To obtain toluene diisocyanate (TDI), often the final product in phosgene-handling plants, the process reacts toluene diamine (TDA) with phosgene, and mixes it with ODB solvent in a reaction tower at high temperature. The outgoing flow of the reaction tower goes to a crude surge tank, and from there it is pumped to a dephosgenation tower (Figure 1) to remove unreacted and excess phosgene from the mixture. This phosgene goes back to the reaction part of the process to be used for further reaction with TDA.

The product of this separation column goes through the distillation and preconcentrating steps to separate the cyanate ion (NCO^-), solvent ODB and any additonal remaining phosgene and other chemicals. The final TDI product leaves the process after separation. The following sections detail the authors' experience in a plant producing TDI using this type of process.

Flow measurement problems

The TDI plant had a problem with flow measurements of crude TDI entering the process. The differential pressure (DP) flowmeter's orifice plate had corroded, and parts of it had been carried away by process flows.

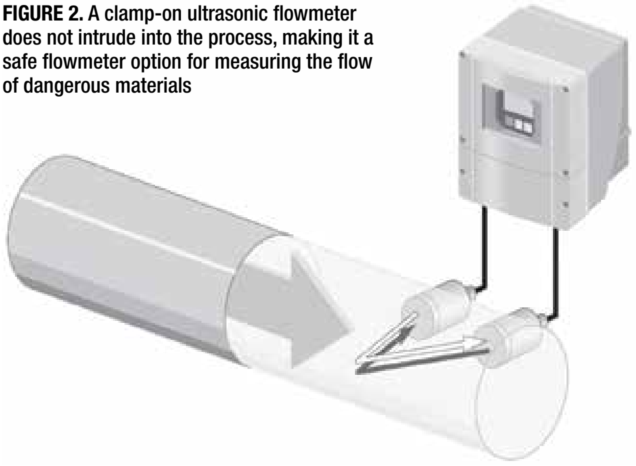
Plant personnel were not able to get the correct differential pressure reading across the orifice taps, resulting in false readings in the transmitter's pressure data when converted to flow. This caused an upset in the process flow from the crude surge tank going to the dephosgenation removal column.

The plant was under severe pressure to not shut down. Production had just started, demand for the product was high, and earlier that year the production unit had come out of a prolonged turnaround due to repair work. The plant produces more than 650 metric tons (m.t.) per day of TDI, resulting in annual sales of more than $400 million, so the need to keep the process running was intense.

The operating company decided to run this part of the process in manual mode based upon historical data from the process historian, but not having realtime flow readings caused process upsets in the distillation column and residue processing. Composition of the residue was not stable and caused residue to polymerize at the operating temperature. To solve this issue, operators had to change the temperature, which caused equipment to run inefficiently.

The distillation column was also unstable and inefficient, requiring more heating and cooling, along

FIGURE 2. A clamp-on ultrasonic flowmeter does not intrude into the process, making it a safe flowmeter option for measuring the flow of dangerous materials



with reflux flow, resulting in excessive energy usage. Several standard operating procedures had to be modified due to the flowmeter problem, so the plant needed a solution — preferably one that would not interrupt operation.

Finding a solution

It was determined that another DP meter would not be suitable. The corrosive nature of crude TDI adversely affected existing orifice plates, and impulse tubing from the orifice plate to the transmitter plugged from crude TDI vapors condensing within. Technicians had to clean impulse tubes twice a year. The tubes are 0.75 in. in diameter and are difficult to clean, and plugging caused errors in the flow reading. It was also a safety issue, as personnel could get exposed to phosgene vapors.

Measuring the flow of crude TDI is not an easy task. TDA, ODB and phosgene react at high temperatures to produce TDI. Therefore, it is a highly heated application with temperatures between 120 and 160ºC (248 and 320ºF), and 90% of the time, the temperature runs around 150ºC (302ºF). Thus, from the reaction tower to the crude TDI tank to the dephosgenation column, the product stays heated by steam, heat tracing and other measures. If crude TDI loses temperature or becomes cold, it can polymerize and plug up the system. This particular process produces 27 m.t./h of TDI, requiring a flowmeter that can measure feed at flowrates up to 700 gal/min.

Because of problems like possible leakage of poisonous gas through flowmeter fittings and flanges, and the corrosive nature of toluene, a flow instrument was needed that would not leak or have threaded connections. With orifice and DP meters, there are several threaded connections, thus introducing the risk of phosgene leakage if those seals and threads fail or become compromised. Therefore, phosgene manufacturers avoid using DP and orifice meters in phosgene service. Phosgene manufacturers worldwide have determined that only three types of flowmeters are suitable for measuring TDI flow: a Coriolis-type mass flowmeter; a vortex flowmeter; and a clamp-on ultrasonic flowmeter.

Coriolis. A Coriolis-type mass flowmeter has several advantages: its measuring principle operates independently of physical fluid properties, such as viscosity or density, and it provides the highest measurement performance for liquids and gases under varying and demanding process conditions, such as temperatures of –50 to 200ºC (–58 to 392ºF). Its accuracy is up to ±0.05% for liquid flow and ±0.1% for volumetric flow.

A Coriolis meter also measures mass flow, density, temperature, volume flow, corrected volume flow, reference density and concentration. The advantages include keeping track of temperature as an additional check, and density can be used to recheck laboratory and analyzer data.

Disadvantages in this application include the requirement of a shutdown or an outage to install the flowmeter, which the plant did not want to do; required piping modifications; the need for an external power supply, which meant running a new cable; and the potential for leakage, causing personnel exposure and environmental safety problems.

Vortex. Advantages of a vortex flowmeter include measurement accuracy of ±0.75% and its ability to operate in temperatures ranging from –200 to 400ºC (–328 to 752ºF). Also, it does not require an external power supply, thus there is no need to run a new cable.

Like the Coriolis meter, these devices require a shutdown or an outage for installation and piping modifications. Other disadvantages include the potential of leakage with attendant safety and environmental problems, and a low cutoff on the flow reading. Low cutoff means that after a certain reading of flow on the low side, the transmitter would read zero. The low cutoff point in this case would have been 40–50 gal/min.

Ultrasonic. The third option, a clamp-on ultrasonic flowmeter (Figure 2), was the solution ultimately deployed at the plant. The measuring principle is independent of pressure, density, temperature, conductivity and viscosity. The measurement accuracy is ±2%, and its temperature range is –40 to 170ºC (–40 to 338ºF), both suitable for this application.

Perhaps most important in this particular application is that this meter did not require a shutdown to install, which meant that the process could keep running. Additionally, it had no potential of developing leaks, thus improving environmental and personnel safety.

Disadvantages included the unavailability of mass and density measurements — but, as the original DP flowmeter did not provide those data either, the plant was able to operate without them. Also, frequent re-gelling where the ultrasonic transducers attach to the pipe is required due to high process temperatures, which cause the gel to dry up.

In this case, the flowmeter also required an external power supply, a new power cable and some piping modifications on insulation. An accessible box was mounted on the pipe so transducers could be accessed for servicing.

To prove the concept, the plant obtained a portable meter to set up a test. Plant personnel removed the insulation from a section of pipe,

FIGURE 3. Ultrasonic transducers are clamped onto the pipe for measuring flowrate

mounted the sensors and holder assembly, and gelled the sensors properly. They then commissioned the transmitter, obtained an acceptable reading, checked it against the data historian, and found that it was within historical recorded flow measurements. Thus, it was determined that the proposed solution would work.

Installation challenges

A commercial clamp-on ultrasonic flowmeter was installed on the line within recommended upstream and downstream pipe-run requirements. Insulation from the piping was removed (Figure 3), and ultrasonic transducers were gelled and clamped onto the pipe. A box was installed around the sensors, as noted above. An external 24-V d.c. power-supply cable was run from a power source to the transmitter. The transmitter was commissioned and began making flow measurements.

The process temperature of around 150ºC (302ºF) was on the high side of the design temperature for the ultrasonic sensors. This required frequent re-gelling of the sensors, otherwise a deviation in the reading would result as the signal from the pipe to the sensors became weak.

An automatic maintenance notification was created in the plant's software to remind maintenance to re-gel the sensors every six months. An appropriate amount of gel was ordered and stored in the spare parts warehouse. After its use, an automatic purchase order could be created by software to order more gel.

A standard operating procedure was drafted to change the mode of flow control to manual during the re-gelling, which would take 15 to 30 minutes; upon completion of re-gelling, it would change the mode back to automatic upon verification of good flow readings.

Another challenge was how to detect drifting in the reading, as there was only one flow sensor. One solution was to install a redundant sensor, but this was too costly. An innovative idea was presented and accepted by operations after computer simulation. Based upon valve opening information, recorded as a percentage, the flow coefficient Cv was calculated based on the valve characteristic and slope equation.

A differential pressure reading was obtained from an available pressure transmitter on the crude TDI surge tank and an available pressure transmitter on the phosgene removal column. From the Cv of the valve and this DP value, the theoretical flowrate was calculated for valve opening in realtime, which was within 5% of the flow reading. An alarm event was programmed in the control system to flag a deviation if it was more than 5% between this virtual flow calculation and the actual flow from the ultrasonic flowmeter.

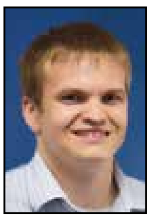
Results were satisfactory, as the ultrasonic flowmeter showed good readings when compared to the historian, and a mass balance simulation verified flow readings were within tolerance. A virtual calculation was also implemented to show drift and deviation as verification.

At this plant that handles phosgene and other dangerous materials, installing a clamp-on ultrasonic flowmeter without shutting down the process allowed the plant to keep operating during installation. This device made the process safer because the non-intrusive ultrasonic flowmeter eliminated any possibilities for leaks of poisonous gas. A thorough evaluation of all available technologies and process-specific requirements are important in selecting the best option for a particular process, especially one that handles extremely hazardous chemicals. ■

Edited by Mary Page Bailey

Authors

Daniel Siddiqui is the U.S. industry manager for chemicals at Endress+Hauser (4333 West Sam Houston Pkwy N., Houston, TX 77043; Email: daniel.siddiqui@us.endress.com), where he oversees marketing for products, training and services related to the chemical industry. Prior to Endress+Hauser, Siddiqui graduated from the University of Houston in 2008 with a degree in electrical engineering. He is a functional safety professional certified by TÜV Germany, and has more than ten years of experience in electrical, instrumentation, automation, advanced process control, functional safety, defect elimination, process improvements, project management and project engineering in the chemical, agricultural and pharmaceutical industries.



Nathan Hedrick holds the position of flow product marketing manager at Endress+Hauser (same address as above; Email: nathan.hedrick@us.endress.com). He has more than seven years of experience consulting on process automation. He graduated from Rose-Hulman in 2009 with a B.S.Ch.E. He began his career with Endress+Hauser in 2009 as a technical support engineer. In 2014, Hedrick became the technical support team manager for flow measurement products, where he was responsible for managing the technical support team covering the flow product line. He has been in the position of flow product marketing manager since 2015.

Liquid-Gas Coalescers: The Effects of Retrograde Condensation

Modeling and pilot testing can minimize its impact during natural-gas processing

Thomas H. Wines and Doug Harris
Pall Corp.
Saeid Mokhatab
Gas Processing Consultant

Retrograde condensation (RC) occurs in multi-component natural-gas streams at relatively high pressures (for instance, pressures above 40 barg/580 psig). The presence of condensed liquids in such systems can have detrimental effects on the performance of liquid-gas coalescer separators, and hence on downstream gas-processing operations. This article reviews several techniques that are available to evaluate retrograde condensation — including both modeling software and pilot testing — and discusses several methods to better control liquid separation and improve process operations.

What is RC?

Retrograde condensation is defined as a "phenomenon associated with the behavior of a hydrocarbon mixture in the critical region wherein, at constant temperature, the vapor phase in contact with the liquid may be condensed by a decrease in pressure; or at constant pressure, the vapor is condensed by an increase in temperature" [*1*].

The term retrograde means contrary to what we would expect. In this scenario, retrograde refers to the fact that the behavior goes counter to what one would normally expect — that is, that under most conditions, lowering the pressure at constant temperature will cause liquids to evaporate, not condense. Retrograde condensation will depend on the composition of the gas, and the temperature and pressure in the system. During the processing of natural gas, retrograde condensation typically occurs at pressures above 580 psig (40 barg) and is very sensitive to the higher-boiling-point components in the gas (C6+ content).

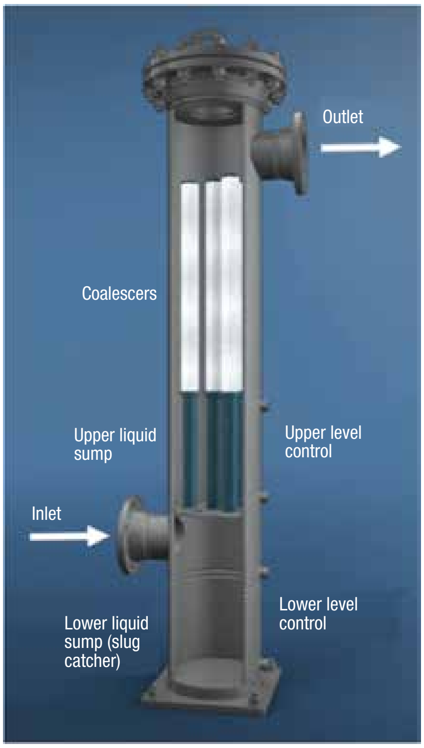


FIGURE 1. Shown here is a typical two-stage, high-efficiency vertical liquid-gas coalescer system that includes an inlet slug catcher

For many liquid-gas separation technologies, the gas flows through a vessel and separation media as a normal course for collecting and removing liquid contaminants. Figure 1 shows a cutaway drawing of a high-efficiency liquid-gas coalescer.

Coalescers will experience pressure drops that are typically in the range of 3 to 15 psid (pounds per square inch differential) over the course of their service life. Pressure drop is caused by inertial losses as a result of the expansion and contraction in the vessel, due to nozzles and the coalescer adaptor connections, as well as from viscous losses from the flow of the gas through the coalescer cartridges. High-efficiency coalescers use a porous medium made from fibrous materials. This medium is designed with fine pores to catch small aerosols down to submicron sizes.

As a result of the pressure drop across the liquid-gas separation system, liquids may be generated if the process is operating in the retrograde region. The use of a surface treatment on high-efficiency vertical liquid-gas coalescers has been found to improve drainage, and thus allow for more compact sizing [*2*]. As retrograde condensation can occur in the coalescer medium wherever there are pressure drops, the coalescer elements will have liquids present throughout the full length of the cartridge during operation. This will affect the sizing of these systems to maintain optimum performance.

Typical problems that can occur

The presence of liquid hydrocarbon condensates that are formed through the retrograde mechanism creates a contamination risk (similar to that posed by any unwanted hydrocarbon liquids that are not effectively separated during gas-processing operations). What makes retrograde condensation especially difficult is that small droplets, which can can form when differential pressure is in the range of just a few psid, can result in additional liquids condensing throughout the plant.

A few processes that can be adversely affected by this phenomenon are discussed in the following section:

Molecular sieves. When molecular sieves are used to remove water vapor from natural gas, liquid hydrocarbons can block off sites, reducing the overall drying capacity of the sieve. Meanwhile, during the thermal-regeneration cycle, heavier hydrocarbons may not desorb quickly

enough, and this can result in fouling or coke buildup on the molecular sieve material, and progressively diminished capacity for water removal and reduced service life [*3*].

Low-NOx burners. Innovation in burner technology has led to several ways to reduce oxides of nitrogen (NO_x) emissions, but such new burner designs typically have smaller orifices that can be more prone to plugging and fouling. Heavy deposits of hydrocarbon-decomposition products may build up on the burner internals, leading to poor flame and heating patterns in the furnace, and may require excessive maintenance to clean fouled burners [*4*].

Gas compressors. Liquid hydrocarbons can affect both reciprocal and centrifugal vane-type compressors. In reciprocating compressors, liquid hydrocarbons can foul the cylinder heads and valves, leading to unscheduled shutdowns for cleaning or repairs. In centrifugal vane-type compressors, deposits of residual heavy hydrocarbons can cause imbalance in the vane rotation leading to vibration and bearing damage [5].

Acid-gas treatment. Liquid hydrocarbons can enter into the alkanolamine (amine) loop that is used to remove either H_2S or CO_2 acid gases from the natural gas stream. This amine is used in a closed, recirculating system that has a contactor to remove the acid gas. Then, on the back side of the process, the amine goes through a regenerator unit, which strips off the acid gas.

For the case of H_2S-containing feed gas, the concentrated acid gas is usually sent to a sulfur-recovery unit. Hydrocarbon condensates may be picked up by the amine, and lighter fractions will leave with the acid gas in the regenerator. This can lead to foam generation in the regenerator, excess air consumption in the sulfur-recovery unit, and possibly tripping the sulfur-recovery unit in extreme cases.

The heavier hydrocarbons may build up in the amine loop and become concentrated over time, leading to fouling of heat exchangers and tower internals, and causing foaming in contactors. This can result in carryover of amine into the downstream gas [*6*].

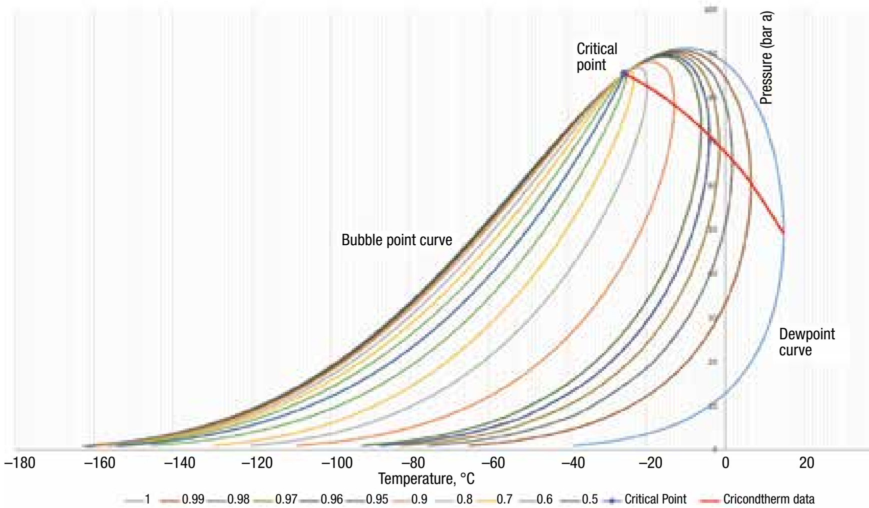


FIGURE 2. This phase envelope, from an offshore platform in Brazil, shows the condensed liquid concentration gradient inside the condensation region the red line is the approximate boundary of the retrograde condensation region. The calculations and diagram were produced using PPDS2 Software and the Physical Properties Database System 2 (PPDS2) / TUV-NEL-UK

Modeling can help

The phase behavior for natural gas with a given composition is typically displayed on a phase diagram, an example of which is shown in Figure 2. The left-hand side of the curve is the bubble-point line. It divides the single-phase liquid region from the two-phase, gas–liquid region. The right-hand side of the curve is the dewpoint line. It divides the two-phase gas–liquid region and the single-phase gas region.

The bubble point and dewpoint lines intersect at the critical point (T_c), where the distinction between gas and liquid properties disappears. The maximum pressure at which liquids can form is called the cricondenbar (P_{cc}), and the maximum temperature at which liquids can form is called the cricondentherm (T_{cc}).

However, there is something interesting within the region $T_c < T < T_{cc}$, while traversing the dewpoint curve from a 0% liquid to another 0% liquid condition during isothermal compression. This cuts through the two-phase envelope, which means that liquid is formed, illustrating the phenomenon of retrograde condensation. It is also important to see that a similar behavior is observed within the region $P_c < P < P_{cc}$. In this case, we experience retrograde vaporization while traversing the bubble curve from a 100% liquid to another 100% liquid condition during isobaric heating.

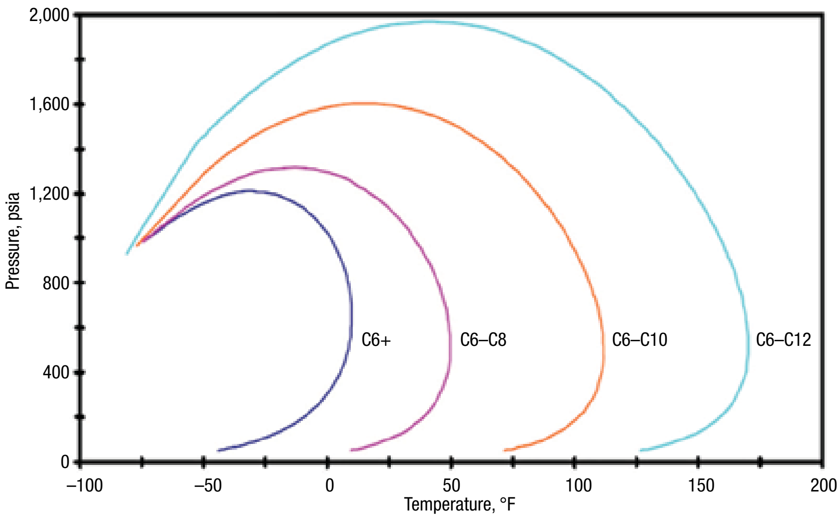


FIGURE 3. Shown here is the effect of C6+ content on hydrocarbon dewpoint [2]

TABLE 1. RESULTS OF PILOT LIQUID-GAS COALESCER FIELD TRIALS AT AN OFFSHORE PLATFORM IN SOUTH ASIA FOR RETROGRADE CONDENSATION				
Case No.	Test flowrate, std. ft^3/min	dP between two housings, psid	Avg. liquid collected on first housing, ppmw	Avg. liquid collected on second housing, ppmw
1	68.4	0	81	None
2	68.4	5	44	1.2
3	96.5	5	92	1.2
4	136.2	5	67.6	2.5
5	136.2	7	77.5	35.2
6	136.2	10	80.3	139.6

The natural gas phase behavior is a function of the composition of the gas mixture, and is strongly influenced by the concentration of the heavier hydrocarbons, especially hexane-plus (C6+). Figure 3 shows the effect of C6+ content on hydrocarbon dewpoint curves. As shown, the presence of heavier hydrocarbons will increase the size of the phase envelope and failure to include them in a phase calculation will under-predict the breadth of the phase envelope. Therefore, there is an essential need for properly characterizing the lumped heavy-end fraction (through C9 or heavier). A complete detailed analysis of the C6+ fraction including all the heavies can be obtained by laboratory gas chromatography (GC) using a representative sample and performing an offsite analysis, as described in Ref. 7.

An accurate knowledge of the behavior of natural gas phases is crucial in designing and operating the gas-liquid separation processes efficiently and optimally. This requires the use of advanced predictive tools for the characterization of hydrocarbon phase behavior, and the best possible wellhead sampling and analysis techniques; These concepts are detailed in Ref. 8.

Gas chromatography (GC) is a common, convenient method used to determine hydrocarbon dewpoint as well as the phase envelope (if desired) for compositional analysis in conjunction with an appropriate equation of state (EOS) [*7*]. The most common EOSs in use in industry today are Soave-Redlich-Kwong (SRK) and Peng-Robinson (PR). Both have been shown to easily and relatively accurately represent the phase behavior in binary and multicomponent systems.

Both SRK and PR give good results in accordance with the experimental data up to about 20 bara. However, at higher pressures, those EOSs tend to under-predict the hydrocarbon dewpoints of a gas stream [*9*]. Generally, at higher pressures, SRK gives dewpoints that are closer to the experimental data compared to the use of the PR methodology. The PR EOS gives a smaller phase envelope than SRK and a good match in the cricondentherm area [*10*]. This effect has been also tested in a research study [*9*] where the authors found that the PR EOS results were in good agreement with the actual hydrocarbon dewpoints, while the SRK EOS predicted dewpoints that were 10–15°F higher than were observed in the experimental tests.

The PR EOS is given below:

$$\left(P + \frac{\alpha a}{\overline{\upsilon}^2 + 2b\overline{\upsilon} - b^2}\right)\left(\overline{\upsilon} - b\right) = RT$$

$$\alpha = \left|1 + (0.37464 + 1.54226\omega - 0.26992\omega^2)(1 - \sqrt{T_r})\right|^2$$

$$\alpha = 0.45724\frac{R^2 T_c^{\,2}}{P_c}$$

$$b = 0.07780\frac{RT_c}{P_c}$$

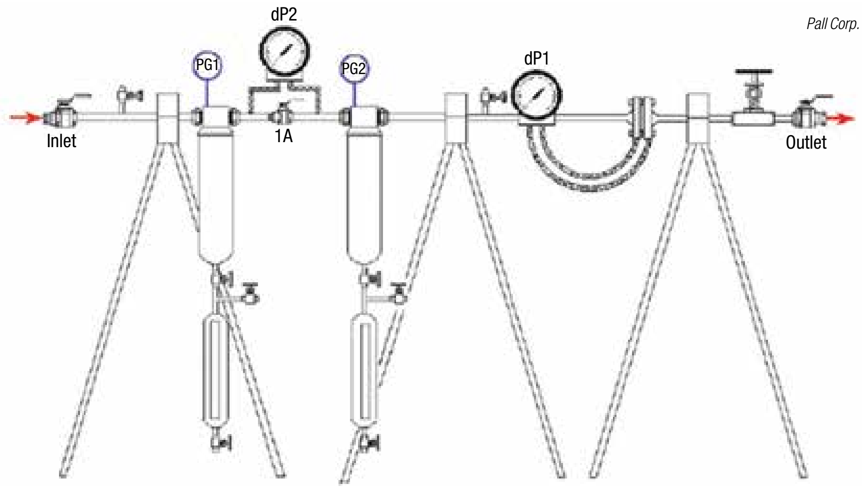
where:
P = pressure, Pa
P_C = critical pressure, Pa
T = temperature, °K
T_C = critical temperature, °K
T_r = reduced temperature, unitless
R = gas constant = 8314.34 M^3-Pa/(kgmol-K)
V = volume, M^3
ω = acentric factor, unitless

Measuring RC in the field

It is possible to run field trials to determine retrograde condensation. Figure 4 shows an apparatus designed for this purpose.

The test equipment consists of two test coalescers (5 in. in length) that are connected in series with a valve (*1A*) in between to create pressure drop. There are isolation ball valves at the inlet and outlet, and a metering valve upstream of the outlet isolation valve that is used to control the gas flowrate. An orifice plate with a DP gage (*dP1*) or other suitable flowmeter is used to measure the flowrate. A second pressure gage (*dP2*) is used to monitor the pressure drop between the two coalescers.

The unit is first run with the valve *1A* open and the amount of liquid collected in the first and second test coalescers are recorded over a period of time — typically a few days. A pressure drop is created across the *1A* valve with increments of a few psid and then the effect of the pressure drop is determined by any changes in the amount of liquids



Pall Corp.

FIGURE 4. Shown here is a gas coalescer system to run field tests for retrograde condensation

BEST PRACTICES

Several best practices for evaluating and mitigating retrograde condensation are as follows:

1. Use Peng-Robinson EOS to evaluate phase envelopes
2. Run a detailed analysis of the heavy hydrocarbon components — at least out to C9
3. Run field evaluations with a two-stage coalescer pilot rig to obtain experimental verification of retrograde condensation
4. If possible, shift the operating parameters (lower pressure or raise temperature) to get out of the retrograde condensation region
5. Size separation equipment more conservatively
   a. Design for low pressure drop to reduce retrograde condensation
   b. For surface-treated, high-efficiency vertical coalescers, model the separation to have liquids wetting out the full length of the cartridges
6. Minimize the distance between separation equipment and downstream process

collected in the second coalescer downstream of the *1A* valve.

The pressure drops created are in the range of 3 to 15 psid to simulate what would happen in a commercial unit. This apparatus is not completely isothermal, so there could be some cooling of the gas due to the Joule-Thompson effect. However, in the pressure drop range of 3–15 psid, this cooling would be minimal. If the test is run in the retrograde condensation region, it will be obvious by the large amounts of liquids that will be generated.

Field tests performed by two of the authors at an offshore platform in Southeast Asia indicated the presence of retrograde condensation with data provided in Table 1. ■

Edited by Suzanne Shelley

References

1. Parker, S.P., "Dictionary of Scientific and Technical Terms," 6th Edition, McGraw-Hill, New York, 2003.
2. Wines, T.H., Improve Liquid/Gas Coalescer Performance, *Hydrocarbon Processing*, 79, 1, 89-96, 2000.
3. Terrigeol, A. and Trifilieff, O., Practical Considerations for the Design of Adsorbent Beds – Molecular Sieve Lifetime Optimization, paper presented at the 23rd Annual Technical Conference of Gas Processors Association - GCC Chapter, Bahrain, Nov. 3-4, 2015.
4. Wines, T.H., Optimize NOx Reductions Facility Wide, *Hydrocarbon Processing*, 83, 4, 53-59 2004.
5. Wines, T.H., High Performance Liquid/Gas Coalescers for Compressor Protection, presented at the 1999 Compressor Work Shop, Lambton College, Sarnia, Ont., Canada, April 28, 1999.
6. Raymond, A., Levesque, F., and Lakhani, H., Separations Technology Improves Amine System's Overall Reliability, *Oil & Gas Journal*, 108, 1, 46-53, 2010.
7. Bullin, J.A., Fitz, C., and Dustman, T., Practical Hydrocarbon Dew Point Specification for Natural Gas Transmission Lines, Research Report 213, GPA Midstream Association, Tulsa, Okla., March 2011.
8. Mokhatab, S., Poe, W.A., and Mak, J.Y., "Handbook of Natural Gas Transmission and Processing," 3rd Ed., Gulf Professional Publishing, Burlington, Mass., 2015.
9. George, D.L., Barajas, A.M., and Burkey, R.C., The Need for Accurate Hydrocarbon Dew Point Determination,*Pipeline & Gas Journal*, 232, 9, 32-36, 2005.
10. Rusten, B.H., Gjertsen, L.H., Solbraa, E., Kirkerød, T., Haugum, T., and Puntervold, S., Determination of the Phase Envelope – Crucial for Process Design and Problem Solving, paper presented at the 87th GPA Annual Convention, Grapevine, Tex., March 2–5, 2008.
11. Dustman, T., Drenker, J., Bergman, D.F., and Bullin, J.A., An Analysis and Prediction of Hydrocarbon Dew Points and Liquids in Gas Transmission Lines, paper presented at the 85th GPA Annual Convention, Grapevine, Tex., March 5–8, 2006.

Authors

Thomas H. Wines is a director of applications development for Pall Corp.'s Fluid Technologies & Asset Protection (FTAP) Group (25 Harbor Park Drive, Port Washington, NY 11050; Email: tom_wines@pall.com). Prior to assuming this role, he was director of product management, senior marketing manager and senior staff engineer for the Scientific and Laboratory Services Division at Pall. Wines has 29 years of filtration, separation, and purification experience serving the refinery, gas processing, and chemical industries, and is a specialist in the fields of liquid-gas and liquid-liquid coalescing. He has authored 47 technical publications and given numerous presentations in this field at professional societies. He holds a Ph.D. in chemical engineering from Columbia University, and is a member of AIChE.

Saeid Mokhatab is an internationally recognized gas-processing consultant based in Halifax, Nova Scotia, Canada (Email: smokhatab@gmail.com), who has worked on the design and operation of several gas-processing plants, and has contributed to gas processing technology improvements through two handbooks and 300 technical papers. Mokhatab has held technical advisory positions for leading professional journals, societies, and conferences in the field of gas processing, and has received a number of international awards and medals in recognition of his work in the natural gas industry.

Doug Harris is sales technical support Leader at Pall Corp. (5 Harbourgate Business, Portsmouth, Hampshire, PO6 4BQ, UK; Email: doug_harris@europe.pall.com). He leads a team of technical inside sales, technical support and applications engineers supporting Pall's outside sales teams and channel partners in the fuels & chemicals, power generation and machinery markets. Harris has worked at Pall for more than 28 years, with roles in Scientific & Laboratory Services, Sales and Marketing, maintaining a focus on the applications of filtration technology in the industrial markets. He holds a bachelor's degree in chemistry from the University of Southampton, and is a member of the Royal Society of Chemistry.

Avoid Safety Pitfalls During Plant Expansion and Modification

Follow this guidance to minimize risk when adding or modifying plant equipment

Bill Wasilewski
Day & Zimmerman
Process & Industrial Group

In 2015, construction in the chemical process industries (CPI) soared. Capital spending surged 18.4% and 255 new chemical production projects were announced, according to the American Chemistry Council (ACC) [*1*]. Since that year, the market has backtracked due, in part, to falling natural gas prices. However, capital spending still increased by 6% in 2017 according to the ACC [*2*]. That means some projects are moving forward. As CPI plant operators consider how to proceed in the coming years, many may opt to increase capacity or yield, or to add new product streams through the use of more modest expansions or upgrades to existing plants, rather than new construction. Plant expansions and renovations can be an effective way to increase capacity without incurring the higher costs associated with grassroots construction.

To make such site improvements as successful and cost-effective as possible, they must be executed carefully, with special attention paid to process engineering and plant safety considerations. Even seemingly minor upgrades can create safety issues if not executed properly. Meanwhile, larger projects may require significant changes to plant layouts and complex engineering adjustments that can make them as challenging from a safety standpoint as greenfield construction.

One of the most famous and unfortunate examples of a plant modification leading to a safety disaster happened in 1974 at a chemical processing plant in Flixborough, U.K. Twenty-eight workers were killed and 36 others were injured in an explosion that was traced back to a modification made two months prior. A leak in one of the plant's reactors had been discovered. To avoid a plant shutdown, engineers installed a temporary pipe intended to bypass the leaking reactor until it was repaired.

A later investigation found that both the pipe that was used and the installation process were substandard. The project was hastily executed without proper consideration for the overall engineering implications. The incident sent shockwaves through the international chemical engineering community, and it led to significant regulatory reforms across Europe, and broader initiatives around the world, focused on improving process safety.

Some of the underlying factors that led to the Flixborough disaster are still a risk today. There will always be pressures on plants to remain efficient and profitable. Communication between contractors and plant operators must always be managed carefully. Despite the numerous process improvements that have been made in the 40 years since this disaster, human error is possible in any complex process.

The engineering upgrade that led to the Flixborough disaster was not a massive expansion of a plant with many moving parts. Rather, the project involved just a single, specific change to one aspect of operations. Today, larger, more complex modifications carry even more risks. To mitigate risk and reduce the incidence of serious hazards, it is critical to examine and understand some of the most common pitfalls that can lead to major accidents at chemical process plants.

Key systems

While managing risk in a plant is complex and involves many moving parts, the equipment systems discussed below are key areas that should be considered most carefully during expansion and modification projects.

Relief-valve systems. Relief valves are essential to maintaining plant safety and avoiding potentially devastating accidents. Unfortunately, proper implementation, and the safe use and functionality of safety valves are sometimes misunderstood or overlooked by plant operators.

When plants expand, the additional throughput capacity that must be handled by the relief valves in the facility usually pushes the limits of

the original design. To compensate, plant operators may need to change out old valves for new ones. In some cases, a valve change is not enough. If a new valve is installed within the same inlet or outlet piping size and configuration, it can create a bottleneck around the new device. Additionally, the relief-collection header system may be inadequate for the new higher pressure levels likely to be encountered. A thorough review of the entire plant is necessary to determine exactly what needs to be replaced and how it should be done.

Some firms may overlook specific relief-valve systems that are not in the immediate vicinity of the new construction. In reality, relief-valve systems in one area of a plant may be impacted by a new piece of equipment hundreds of yards away. If any single relief valve is not adequate or does not meet proper specifications it could lead to a complete disaster.

On occasion, plant operators also overestimate the capabilities of a particular piece of equipment. For instance, a boiler might be capable of handling an increased amount of pressure as the result of a new project. But there may be piping or valves attached to that boiler that are not able to handle the increased pressure.

A complete audit of relief-valve systems should be a part of any plant modification, expansion or update. It is critical that plant operators partner with contractors to ensure that no part of this process is overlooked or rushed. Problems with relief valves can be one of the most devastating and dangerous problems a chemical process plant can face.

Piping and pipe racks. In addition to relief valves, piping should be thoroughly assessed and reviewed during any type of plant modification or expansion. In a mature plant, pipe racks often have little or no space for expansion. These limitations should be identified early in the planning process so that there are no unexpected costs for rack expansions. It's also not uncommon during modifications to discover that pipe racks are overloaded beyond their design rating. This poses serious safety risks that could lead to potential explosions. That's why it is always more prudent to add additional pipes to increase flow.

Another potential hazard is the presence of "dead-leg" pipes. These pipes are disconnected from the plant's process and are left behind from previous maintenance work or plant fixes. Dead-leg pipes take up unnecessary space on a pipe rack, but more importantly, they pose a safety risk. Stagnant liquids left in dead-leg pipes can lead to pipe corrosion or rupture if the liquid freezes.

Many plants have dead-leg pipes that have not been part of the active process for years. As dead legs increase in number, the risk to the plant multiplies with them. When dealing with hazardous chemicals, small amounts of pooling liquid in dead legs can lead to major ruptures or leaks. Plants should have regular programs in place to inspect pip-

ing. While there are scenarios where it may make sense for a dead-leg to remain, unnecessary dead-legs should be removed during plant expansions and modifications.

If there is a change in plant processes during construction, it could require further changes in the piping. For instance, new chemicals may require different piping materials. A process change may also require new gaskets or elastomers.

Piping and pipe racks are everywhere in chemical process plants. It only takes a problem arising in one of those pipes for a safety incident to occur. That's why a thorough inspection of pipes and pipe racks is necessary as part of any expansion or modification.

Access and confined-space issues. Adding new pieces of equipment or building new areas onto an existing plant can have a significant impact on plant layout. Large vessels installed to increase output, can also create new hazards. Placement of new equipment may create unsafe confined spaces or limit access to critical infrastructure. While a certain number of confined spaces in a CPI plant may be unavoidable, designs should try to minimize them as much as possible.

During construction, there are often temporary installations that restrict access to certain areas of the plant. Any additional equipment that is necessary to complete a project — such as scaffolding — should be placed carefully to ensure that it does not restrict key access areas.

Even under the best of circumstances, confined spaces are extremely dangerous. Potential safety hazards associated with confined spaces include the ignition of flammable liquids or gases, asphyxiation and exposure to hazardous chemicals [*3*].

The U.S. Occupational Safety and Health Admin. (OSHA) has strict standards for confined-space safety. Workers are not allowed to enter confined spaces unless they have proper training and specific equipment. But despite these safety standards, accidents happen with troubling regularity. The U.S. Bureau of Labor Statistics estimates that on average there are 92 fatal injuries in confined spaces every year.

Appropriate labeling (especially for chemicals and piping). Having proper equipment labeling and documentation is the easiest and the least costly way to improve safety. It is also one of the most overlooked. According to OSHA, hazard communication is the second-most-cited safety violation for businesses, behind fall protection [*4*]. Last year, OSHA began requiring all chemical-hazard labels to adhere to the Globally Harmonized System established by the United Nations. This system was designed to be universally understandable.

While OSHA has made great strides in the area of chemical labeling, it does not mandate every type of labeling in a plant. Particularly when it comes to piping, the responsibility for labeling falls solely on the plant operator. While there are no specific OSHA requirements for pipe labeling, OSHA does recommend the ANSI/ASME A13.1 pipe-marking standard [*5*].

Even though OSHA does not have strict requirements, plants should follow a prudent approach — those that don't label pipes do so at their own peril. New construction ofen changes equipment processes and therefore labels should always be updated to reflect changes, so there is no confusion.

To make equipment upgrades and site improvements as successful and cost-effective as possible, they must be executed carefully, with special attention paid to process engineering and plant safety considerations. Even seemingly minor upgrades can create safety issues if not executed properly.

Closing thoughts

When a plant adds new features, installs new capital-intensive equipment or expands production capacity, it is an exciting time for plant operators, workers and the community. It means increased revenue for the company, jobs for workers, and economic development for local communities. While plant operators want to be efficient with their capital spending, many of the potential pitfalls discussed here are not costly to investigate and correct. Plant operators should find engineering, design and construction partners that are committed to thoroughly evaluating all of these areas to ensure that their investments are protected and are not put in peril. ■

Edited by Suzanne Shelley

Author

Bill "Waz" Wasilewski is president of process and industrial for Day & Zimmermann (Patewood 5, 80 International Dr., Suite 100, Greenville, S.C.; Email: Bill.Wasilewski@dayzim.com). He is responsible for oversight of the company's chemical, advanced manufacturing, petroleum refining, and food and beverage customers. He has more than 30 years of experience at large engineering and construction firms.

References

1. Year-End 2015 Chemical Industry Situation and Outlook, American Chemistry Council, www.americanchemistry.com/Jobs/EconomicStatistics/Year-End-2015-Situation-and-Outlook.pdf
2. Outlook: U.S. Manufacturing Turns Corner, Rides Upswing of Global Economies; Chemical Industry Growth Rate Surpasses 20-Year Average as Industry Rebounds from Historic Hurricanes, American Chemistry Council, www.americanchemistry.com/Media/PressReleasesTranscripts/ACC-news-releases/Outlook-US-Manufacturing-Turns-Corner.html
3. Meyers, Kelly, 7 Safety Incidents You Should Look Out For in the Oil & Gas Industry, Blackline Safety, www.blacklinesafety.com/7-employee-safety-incidents-you-shouldnt-overlook-in-the-oil-gas-industry/
4. Top Ten Most Frequently Cited Standards, Occupational Safety and Health Administration, www.osha.gov/Top_Ten_Standards.html
5. Graphic Products Editorial Staff, OSHA Pipe Labeling Requirements, Graphic Products, www.graphicproducts.com/articles/osha-pipe-labeling-requirements/

Biosludge Drying: System Design Schemes for the Sticky Phase

Drying biosludge from wastewater-treatment plants is complicated by the "sticky phase," making the practice more of an art than a science. Discussed here are efforts to define the beginning and endpoints of this phase analytically

Yen-Hsiung Kiang
Xiamen Baipeng Environmental Engineering Co. Ltd.

The drying of biosludge from a wastewater-treatment plant (WWTP) is not an easy task. The most troublesome problem during biosludge drying is caused by the so-called sticky phase of the biosludge. This phase is characterized as a partially dewatered, paste-like material that adheres to the surface of the drying equipment. Mostly because of the difficulties associated with handling the sticky phase, the drying of biosludge is currently thought of as somewhat more of an art than a science. This article represents an effort to move this drying activity further into the realm of science, by developing an analytical approach to defining the beginning and ending solid concentrations for the sticky phase zone of biosludge.

By using laboratory test data from a previous article on this topic (*Chem. Eng.,* September 2014, pp. 51–54) as an example, this article presents design schemes for biosludge drying that can make use of these predicted beginning and ending concentrations of the sticky phase zone. Armed with the information presented in this article, designers and operators of wastewater biosludge drying systems can set up drying systems with higher efficiency and fewer problems associated with the sticky phase.

Sticky-phase zone boundaries

Biosludge normally contains high water content. The heating values are normally low and the material is difficult to use directly as a fuel. Thus, to use biosludges as fuel, water must be removed before feeding the material into combustion equipment.

During the dewatering and drying process, the wastewater sludges, pass through what has come to be called the sticky-phase zone. Although this stage of property change has been observed for a long time, it was first quantitatively characterized by Peeters and others [*1*]. This line of research utilized a shear-test-based laboratory protocol to map out the changes in sludge properties during the drying process. For more information about mapping the stickiness during biosludge drying, see Ref. 2.

This article discussed five different sludge samples that were used for the testing. These samples were taken from the Monsanto WWTP in Antwerp, Belgium. The organic content of the sludge samples varies from 49 to 30 wt. %. The testing data presented by Peeters show that the sludge's sticky characteristics depend on both the sludge's water content during drying, and on the initial organic content. A summary of the data from the Peeters study is shown in Table 1.

TABLE 1. STICKY PHASE RANGES DURING SLUDGE DRYING FOR FIVE SAMPLES (DATA SOURCE: [*1*])

Sludge sample	Organic material in the solids, %	Water content, wt. %	
		Sticky phase beginning	Sticky phase end
1	49	76	62
2	47	76	62
3	35	65	42
4	35	64	45
5	30	55	42

Based on these data, the sticky-phase zone appears in a particular water content range. However, the exact range of the sticky-phase zone is dependent on the properties of the sludge. It is interesting to note that the lower the organics content in the solid faction of the sludge, the lower the water content of sludge where the sticky phase begins. The reason that drying sludge forms a sticky phase is that during biotreatment, microorganisms are entrapped within the activated sludge, and they form long-chain biopolymers [*2*]. The biopolymers "glue together" the microorganisms in the sludge. During dewatering and drying, the gluing biopolymers become more and more concentrated and result in the formation of a sticky mixture.

Phases of the sticky-phase zone

In general, the stickiness property zones for individual sludge samples can be qualitatively summarized by the diagram shown in Figure 1, which shows the three sludge property phases commonly observed during drying of biosludge. The three stages are described further here:

1. At high water content (low solids), the wet sludge does not behave as a sticky substance because at that stage, it is a biopolymer solution with low concentration.
2. When the sludge's water content decreases (and dry solid content increases), the biopolymer solution becomes more and more concen-

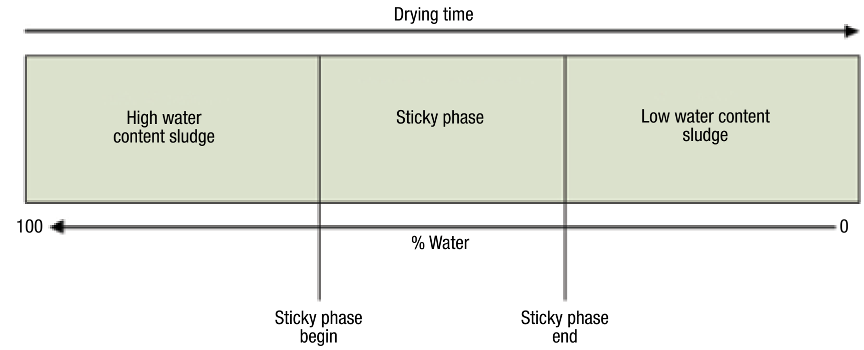


FIGURE 1. The stickiness properties of drying sludge from wastewater treatment plants is divided into three general zones

trated, and the overall biosludge becomes increasingly sticky, until it reaches the sticky-phase zone. The concentration and range of the sticky phase are sludge-property specific.

3. As the water content is lowered further in the drying process, just beyond the range of the sticky phase zone, the sludge abruptly decreases in stickiness. The reason is that, at this point, the biopolymers are dry enough to be dispersed without sticking together.

The three phases of the sludge drying process are shown in real-world photographs in Figure 2. These pictures are taken from a sludge-dryer operation film for a print shop wastewater sludge [*3*].

Sticky-phase mechanisms

Why does biosludge with a lower organic content have its sticky phase zone form at lower water contents? Lower organic content of the biosludge (as a result of a temporarily higher amount of precipitated calcium carbonate salts in the sludge floc because of changing wastewater composition, for example [*4*]), implies a lower level of extracellular polymeric substances per unit of sludge mass. Thus, the content of extracellular polymeric substances determines the sticky-phase range of the sludge.

Predicting start and endpoints

The starting and ending water concentrations of the sludge's sticky-phase formation zone during sludge drying can be predicted by two models. These models aim to take some guesswork out of predicting the start and endpoints of the sticky-phase formation zone.

These two models were developed by regression analysis, based on the data in Table 1. The range of the organics content in the dry solids is between 30 and 49 wt.%.

Model S101. This model can be used to estimate the beginning concentration of the sticky-phase zone during biosludge drying:

$$\%C_h = -62.6563 + 5.638979\ \%C_o - 0.0573\ \%C_o^2 \quad \textbf{(1)}$$

Model S102. This model is intended to estimate the ending concentration of the sticky-phase zone during biosludge drying:

$$\%C_l = 74.707 - 2.5054\ \%C_o + 0.04655\ \%C_o^2 \quad \textbf{(2)}$$

Where

$\%C_h$ is the percent water content by weight at the beginning of the sticky-phase zone

$\%C_l$ is the weight percent water content at the end of the sticky-phase

$\%C_o$ is the percent organic materials content by weight of the biosludge in the feed to the sludge dryer

The correlation coefficients are 0.9983 for Model S101 and 0.9699 for Model S102. The estimation errors are less than ±1% for Model S101 and less than ±5% for S102.

Drying-system design schemes

During the equipment design phase for a sludge-drying process, there are many approaches that can be used to overcome the problems introduced by the sticky phase of drying sludge. The final choice is dependent upon the requirements of a particular sludge-drying application.

Presented here are five possible strategies for addressing the problems associated with the sticky phase that can be informed by identifying the starting and ending points of the sticky phase, as discussed earlier. The five strategies are the following:

Scheme A. Dry the sludge to a point before the sticky phase begins. If the purpose of the sludge drying is only to reduce the weight and vol-

ume required for transportation and disposal of the sludge, the sludge should only be dried to the point before the sticky phase begins.

The sludge-drying reduction ratio (weight), which is represented by the wet sludge input rate over the dry sludge output rate, can be calculated as follows:

$$R = (100 - \%W) / \%D \qquad \textbf{(3)}$$

Where
R is the reduction ratio (weight), wet sludge input rate over dry sludge output rate
$\%D$ is the weight percentage of dry solids content in the wet sludge
$\%W$ is the weight percentage of water content in at the beginning of sticky phase zone

For example, for sludge Sample No. 5 in Table 1, when drying the 80%-water-content sludge to the beginning of the sticky phase begins, water content is 55%. The reduction ratio is around 2.25. After the sludge in this sample is dried, the required transportation and disposal is reduced by around half.

Scheme B. Design the drying equipment with higher torque and sturdy design. This approach may be the most expensive method, but it can resolve the root of the problem.

Scheme C. Back-mix dry sludge products into the wet sludge being fed into the dryer, such that the water content of the mixed sludge fed into the dryer is below the ending concentration of the sticky phase. This approach will increase the required size of the sludge dryer. The system is shown in Figure 3.

The ratio between the dry-sludge recycle rate and the wet-sludge feed flow rate (weight) can be calculated as follows:

$$R = (W1 - X) / (X - W2) \qquad \textbf{(4)}$$

Where
R is the ratio between the recycle dry sludge flowrate and the wet sludge feed flowrate (weight)
$W1$ is the water content of the wet sludge input
$W2$ is the water content of the dry sludge output
X is the water content of the sludge at the end of the sticky phase zone.

Scheme D. Control the dryer environment in such a way that inside the dryer, the water content of the sludge to be dried is always lower than the ending concentration of the sticky point zone. This approach can be achieved by first filling the dryer chamber with already dried products. Then, wet sludge is fed into the dryer in small quantities at a time, so that the wet sludge feed mixes with the dried products readily for a mixed bulk sludge. This bulk sludge has a water content inside the dryer that is always less than the endpoint concentration of the sticky-phase zone.

Scheme E. Introduce additive, polyaluminum chloride (PACl), into the wet sludge feed to alleviate, or eliminate, if possible, the stickiness of the sludge [*5–7*] The possibility of using PACl for the adjustment, or conditioning, of wet sludge is that the bound hydration water within the super-aluminum structures of

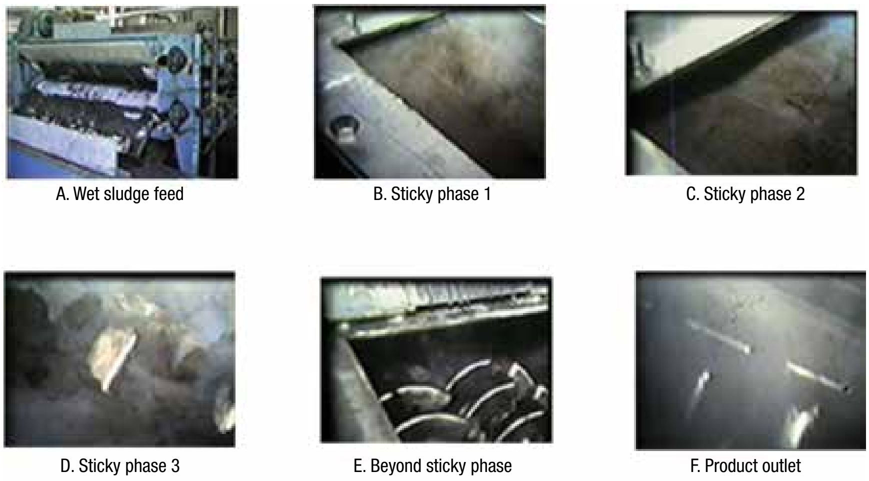


FIGURE 2. The series of photos shows an example of sludge passing through the sticky phase in a drying process

the PACl solutions will adhere to the exterior of the sludge flocs. And, the hydration water acts as a lubricant to channel the sticky biopolymers through the dryness range [*7*].

Concluding remarks

The models for the sticky-phase of drying WWTP sludge provide an estimate of the beginning and ending water concentrations of the sticky-phase. These models represent an initial effort to define the range of the sticky zone more scientifically than has been possible in the past.

By making use of these predicted beginning and ending concentrations of the sticky phase zone, the designers and operators of wastewater biosludge drying systems can avoid some of the problems observed with the sticky phase and can develop drying systems with higher efficiencies. However, in order to make the models more useful, much more work is required to expand the data for the beginning and ending of the sticky-phase zones. The data used in this article are based on samples from the Monsanto WWTP in Antwerp, Belgium, but a wider range of samples are recommended to widen the applicability of the models to biosludges from different sources and regions. This work will help to improve the prediction models. ■

Edited by Scott Jenkins

Acknowledgment

Some materials are taken, with permission, from "Fuel Property Estimation and Combustion Process Characterization," by Yen-Hsiung Kiang, published in 2018 by Academic Press. This research did not receive any specific grant funding.

References

1. Peeters, Bart, Dewil, Raf, Van Impe, J.F.M., and Smets, Ilse, Using a shear test-based lab protocol to map the sticky phase of activated sludge, *Environmental Engineering Science* 28, pp. 81–85, 2011.
2. Peeters, Bart, Dewil, Raf, Van Impe, J.F.M. and Semts, Ilse, Challenges of Drying Sticky Wastewater Sludge, *Chem. Eng.*, September 2014, pp. 51–54.
3. Kiang, Yen-Hsiung, information from the author's personal collection.
4. Peeters, Bart, Dewil, Raf, Lechata, D. and Smets, Ilse, Quantification of the exchangeable calcium in activated sludge flocs and its implication to sludge settleability, *Separation and Purification Technology*, 83, pp. 1–8, 2011.
5. Peeters, Bart, Dewil, Raf, Vernimmen, Luc and Smets, Ilse, Avoiding sludge stickiness through addition of polyaluminium chloride (PACl), In: Proceedings of the 11th World Filtration Congress (WFC11), Graz, Austria, paper P368, April 16–20, 2012.
6. Peeters, Bart, Dewil, Raf, and Smets, Ilse, Improved process control of an industrial sludge-dryer installation through binary logistic regression modeling of the fouling issues, *J. Process Control*, 22, pp. 1,387–1,396, 2012.
7. Peeters, Bart, Dewil, Raf, Vernimmen, Luc, Van den Bogaerta, Benno and Smets, Ilse, Addition of polyaluminium chloride (PACl) to waste activated sludge to mitigate the negative effects of its sticky phase in dewatering-drying operations, *Water Research*, 47, pp. 3,600–3,609, 2013.

Author

Yen-Hsiung Kiang is the chief technology officer of Xiamen Baipeng Environmental Engineering Co., Ltd. (16F, Yong Tong Chang Building, Jiahe Rd., Xiamen, 36100, China; Email: yenhk168@yahoo.com; Phone: +86-1521-0324628). Kiang has over 40 years experience in fuels, incineration, combustion, solid waste, sewage sludge drying, composting, groundwater pollution, soil contamination, cogeneration and liquefied natural gas, wastewater treatment, air pollution, and other areas. He received his M.E. and Ph.D. in chemical engineering from the University of Florida, and is a registered professional engineer in Pennsylvania. Kiang is a member of the boards of directors of Falcon Power Co., Ltd. and China Alternative Energy Association, as well as a consultant to the Energy and Carbon Reduction Office of Executive Yuan, ROC and lead consultant of Sustainable Development Company, Ltd. Throughout his career, he has executed planning and design of hundreds of waste treatment centers (including incineration, cogeneration, physicochemical treatment, solidification and landfill).

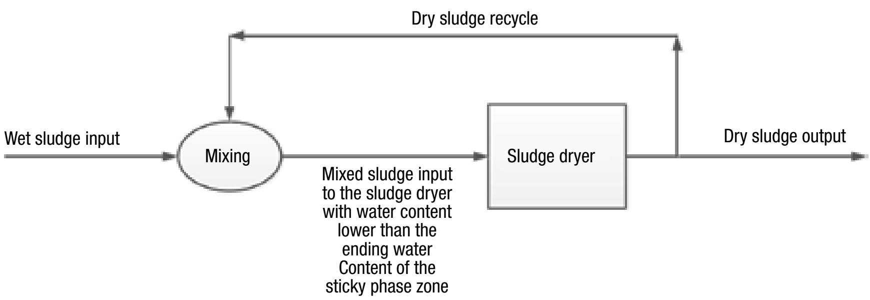


FIGURE 3. The drying strategy discussed in Scheme C, involving back-mixing of dry sludge, is shown in this diagram

Economic Indicators

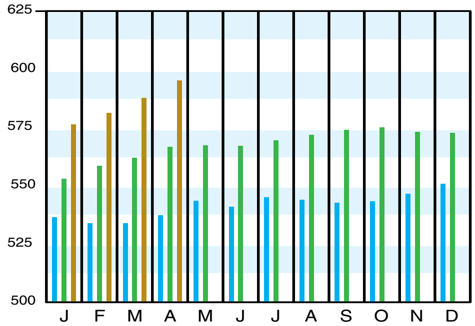
2016 2017 2018

Download the CEPCI two weeks sooner at www.chemengonline.com/pci

CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957–59 = 100)	Apr. '18 Prelim.	Mar. '18 Final	Apr. '17 Final
CE Index	595.3	588.0	566.6
Equipment	723.8	713.3	684.2
Heat exchangers & tanks	637.5	626.0	600.8
Process machinery	710.7	703.4	673.0
Pipe, valves & fittings	952.3	930.4	885.0
Process instruments	419.4	417.9	404.2
Pumps & compressors	1015.6	1017.7	978.6
Electrical equipment	533.2	532.8	515.5
Structural supports & misc.	776.0	763.3	735.7
Construction labor	331.7	331.3	324.0
Buildings	586.4	582.1	556.5
Engineering & supervision	310.9	310.0	314.2

Annual Index:
2010 = 550.8
2011 = 585.7
2012 = 584.6
2013 = 567.3
2014 = 576.1
2015 = 556.8
2016 = 541.7
2017 = 567.5

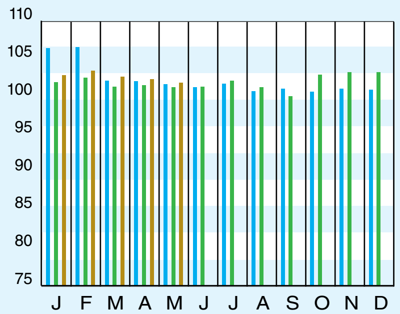


Starting in April 2007, several data series for labor and compressors were converted to accommodate series IDs discontinued by the U.S. Bureau of Labor Statistics (BLS). Starting in March 2018, the data series for chemical industry special machinery was replaced because the series was discontinued by BLS (see *Chem. Eng.*, April 2018, p. 76–77.)

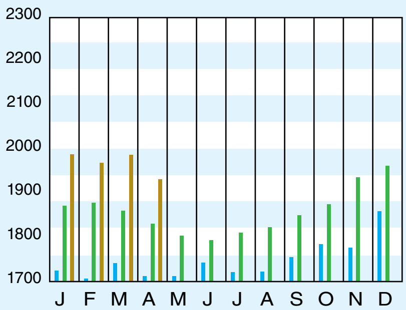
CURRENT BUSINESS INDICATORS

	LATEST	PREVIOUS		YEAR AGO
CPI output index (2012 = 100)	May '18 = 101.8	Apr. '18 = 102.1	Mar. '18 = 101.7	May '17 = 99.9
CPI value of output, $ billions	Apr. '18 = 1,932.4	Mar. '18 = 1,934.7	Feb. '18 = 1,919.8	Apr. '17 = 1,762.4
CPI operating rate, %	May '18 = 75.8	Apr. '18 = 76.1	Mar. '18 = 75.9	May '17 = 75.3
Producer prices, industrial chemicals (1982 = 100)	May '18 = 267.7	Apr. '18 = 265.8	Mar. '18 = 269.1	May '17 = 253.5
Industrial Production in Manufacturing (2012 =100)*	May '18 = 103.5	Apr. '18 = 104.2	Mar. '18 = 103.6	May '17 = 101.8
Hourly earnings index, chemical & allied products (1992 = 100)	May '18 = 185.2	Apr. '18 = 187.3	Mar. '18 = 190.0	May '17 = 174.7
Productivity index, chemicals & allied products (1992 = 100)	May '18 = 96.7	Apr. '18 = 96.2	Mar. '18 = 97.1	May '17 = 98.8

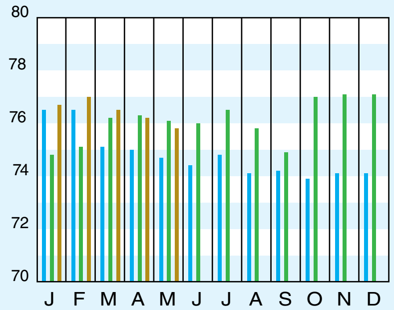
CPI OUTPUT INDEX (2000 = 100)†



CPI OUTPUT VALUE ($ BILLIONS)



CPI OPERATING RATE (%)



*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board.
†For the current month's CPI output index values, the base year was changed from 2000 to 2012
Current business indicators provided by Global Insight, Inc., Lexington, Mass.

CURRENT TRENDS

The preliminary value for the April 2018 *CE* Plant Cost Index (CEPCI; top; most recent available) jumped compared to the previous month's value, continuing a string of monthly increases since the beginning of the year. And for the third consecutive month, all four of the subindices (Equipment, Buildings, Construction Labor and Engineering & Supervision) moved higher for April. The Equipment subindex had the largest month-to-month increase, percentage-wise. The overal CEPCI for March stands at 5.1% higher than the corresponding value from April of last year. Meanwhile, the Current Business Indicators (CBI; middle) showed a small decrease in the CPI output index for May 2018.